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SEMIMICROQUANTITATIVE ANALYSIS OF COPPER 8-QUINOLINOLATE

A Demonstration of Uneven Deposition of Fungicide on Cellulosic Materials as a Major Variable in Deterioration Evaluation

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INTRODUCTION

FUNGICIDES analyzed by methods affording reproducible results have been found to be present in textiles in biologically effective concentrations even though loss in breaking strength was considerable after mildew resistance test. This apparent anomaly has been ascribed consistently to variations inherent in biological tests, particularly soil burial. Biological methods in these laboratories have been found to be highly reproducible and therefore diagnostic of potential mildew resistance for military materiel and equipage. Accordingly, critical evaluation of data showing lack of agreement between breaking strength and chemical analysis suggested as sources of variation the measurement of breaking strength and the chemical analytical methods as given.

The measure of breaking strength was experimentally determined in this laboratory (unpublished data) to be as adequate as more refined physical tests such as the calculation of energy absorption from planimetric measure of stress-strain curves and elongation. Chemical methods requiring analysis of relatively large sample weights were suspected, therefore, as a possible cause of the lack of agreement of fungicide content with loss of breaking strength after soil burial. In the event of uneven deposition of the fungicide on a thread or textile this variation would be reduced by the large size of the sample analyzed. The chemical method could determine, therefore, only the average concentration of the mass analyzed. Thus reproducible chemical analyses would be obtained which would not reflect evenness of deposition of the fungicide as effectively as the biological methods mentioned previously.

In the case of 12/4 cotton sewing thread, for example, 300 to 400 mg of thread is the quantity analyzed routinely in this laboratory for copper 8-quinolinolate by the method of Rose et al *(11)*. This weight of thread is approximately seven feet long. Obviously, no conception of deterioration relative to variation in amount of copper 8-quinolinolate deposited on the thread can be obtained with this size of sample. The potential inadequacy of chemical analytical methods remained unknown as to their capacity to reflect the changes in susceptibility of mildewproofed materials to biological degradation.

Therefore, the need to achieve adequate semimicroquantitative chemical analytical methods was apparent in order to determine the extent of variation (if it existed at all) in the amounts of copper 8-quinolinolate deposited. This variation between analysis of fungicide content and breaking strength could be relevant to all fungicides applied to cellulosic materials. Its pertinency with regard to thread and textile mildewproofed with copper 8-quinolinolate was investigated first. These studies are the subject of this report.

A rapid and relatively simple procedure would be most desirable because it would be used routinely in the analysis of cotton materials treated with copper 8-quinolinolate. These requirements suggested a method based on colorimetric or spectrophotometric measures. Since concentrations as low as 1γ metallic copper as copper 8-quinolinolate might have to be analyzed, only the more sensitive reagents adapted to colorimetric or spectrophotometric instrumentation were considered. With some modification, a semimicroquantitative method was adapted from the procedure for analysis of copper 8-quinolinolate described by Rose et al *(11)* that correlated well with large sample analysis and with a sensitive but more laborious method for copper proposed by Woelfel *(13)*. The method of Rose et al affords analysis of copper 8-quinolinolate directly in contrast to that of Darbey *(2)*, which determines spectrophotometrically the 8-hydroxyquinolinium ion as a measure of copper 8-quinolinolate. Moeller *(6)* reported extensively on the spectrophotometric properties of copper 8-quinolinolate. Other methods described for copper analysis report the use of rubeanic acid *(1, 10)* dithizone *(3, 4, 8)*, sodium diethyldithiocarbamate *(5)*, dithiocarbamate and salicylate *(7)*, O-nitrosophenol *(9)*, and benzo fast yellow *(12)*.

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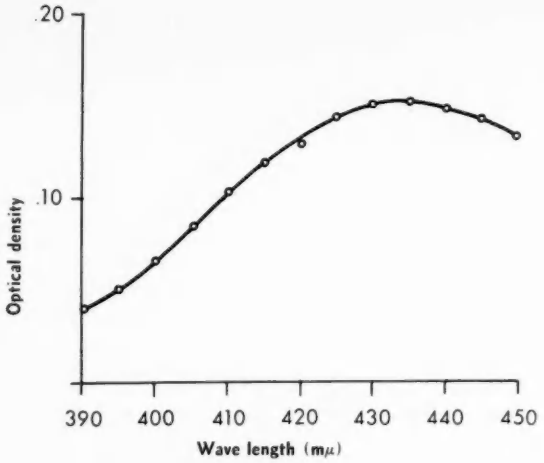


Figure 1
Spectrophotometric curve for copper diethanoldithiocarbamate concentration of copper = approximately 0.05 mg/100 ml solution.

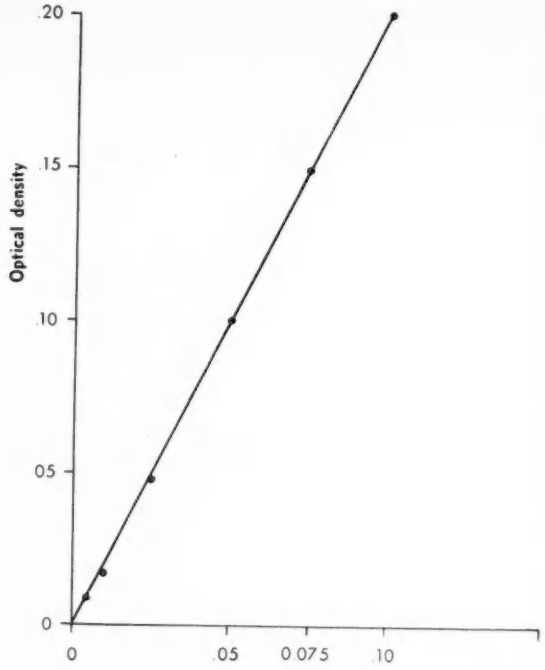


Figure 2
Standard curve for copper measured spectrophotometrically as copper diethanoldithiocarbamate. Readings made at 434 mμ.

PROCEDURE

ADAPTATION OF THE SPECTROPHOTOMETRIC METHOD OF ROSE ET AL *(11)*———Weigh to the fifth place on a semimicro balance three to four mg of material (approximately one inch of a 12/4 thread) and place in a 10-ml Erlenmyer flask. Extract the material at 70° to 80° C first with three ml of 10 percent H_2SO_4 to which is added two drops of $CHCl_3$, then with two ml of 10 percent H_2SO_4 to which is added two drops of $CHCl_3$, and last with two ml of 10 percent H_2SO_4 diluted with three ml distilled H_2O. Heat the first two extractions until the odor of $CHCl_3$ is no longer detectable (about 10 minutes) and the last extraction for about five minutes. Decant each extract into a 25-ml beaker. Adjust the pH of the combined extract to 6.5 potentiometrically with concentrated NH_4OH. Transfer the solution to a 60-ml separatory funnel and extract successively with four-, three-, and three-ml portions of $CHCl_3$. Collect all $CHCl_3$ extractions in a beaker containing anhydrous Na_2SO_4. Transfer the contents of this beaker quantitatively to a No. 40 Whatman filter and collect the filtrate in a 10-ml volumetric flask. Wash the residue with $CHCl_3$, collecting the filtrate to the 10-ml mark on the flask. Read the optical density of the solution at 410 mμ using one-cm Corex cells in a Beckman DU spectrophotometer. $CHCl_3$ is used as the blank in the reference cell. Determine milligrams of copper 8-quinolinolate from the standard curve and calculate the percent of the compound from the following equation:

$$\text{Percent copper 8-quinolinolate} = \frac{(\text{mg Cu-8 / 100 ml } CHCl_3 \div 10)}{(\text{mg wt of sample})} \times (100)$$

The standard curve established in accord with the procedure proposed by Rose et al *(11)* is suitable for this semimicro adaptation. This is so because the optical density of small amounts of copper 8-quinolinolate is maintained by reduction of the volume. The factor of $\frac{1}{10}$ in the above equation for percent copper 8-quinolinolate corrects the determination in accord with the volume prepared.

MODIFICATION OF WOELFEL'S METHOD II *(13)* FOR SEMIMICRO-QUANTITATIVE DETERMINATION OF COPPER AS COPPER 8-QUINOLINOLATE———The procedure developed in this laboratory followed that reported by Woelfel *(13)* except that a spectrophotometer to measure absorbence was used instead of the filter photometer recommended. The curve obtained with a Beckman DU spectrophotometer betwen 390-460 mμ using a solution containing 0.05 mg copper as copper 8-quinolinolate per 100 ml showed an absorbency peak at 434 mμ (Fig 1). At this wave length Beer's law was found to hold for solutions containing up to 0.10 mg copper per 100 ml (Fig 2). Higher concentrations were not studied.

The procedure in detail follows:

Digest three to four mg of material weighed on a semimicro balance to the fifth place in a micro-Kjeldahl flask with 0.4 ml 1:1 H_2SO_4. Cool and add a few drops of 30 percent H_2O_2 to hasten the oxidation. Heat and cool the digest and add the peroxide alternately until the oxidation is complete. Dilute the digest to 10 ml with double-distilled water. (Note: Distilled water gave an optical density of 0.043 compared with 0.008 for double-distilled H_2O). Add 1.2 ml of 12 HNO_3 and neutralize with 15N NH_4OH. Add to the neutral solution one drop of 1.2N HNO_3 to make the solution just acid to litmus. Transfer the solution to a 25-ml volumetric flask. Add 2.5 ml of acetate buffer and 0.25 ml of Cuprethyl reagent [diethanolammonium bis (2-hydroxyethyl dithiocarbamate)] and dilute to mark with double-distilled water. Optical density readings are then made at 434 mμ using a Beckman DU spectrophotometer and the solution prepared from the extract of untreated material as the blank in the reference cell. The standard curve is prepared by measuring the optical density of known weights of copper as copper 8-quinolinolate treated as outlined above with double-distilled water in the reference cell.

RESULTS AND DISCUSSION

ANALYSIS OF THREAD TREATED WITH COPPER 8-QUINOLINOLATE——Initial analysis of thread using the semimicroquantitative adaptation of the method of Rose et al *(11)* included 12 samples ranging in weight from 3.10 to 4.12 mg. The average concentration was found to be 0.88 ± .03 percent for thread treated to contain nominally 1.0 percent copper 8-quinolinolate. The individual determinations ranged from 0.63 to 1.03 ± .09 percent of the compound. The determinable error in the weighing of the sample was 0.05 mg per 4.50 mg (one percent) and 0.002 per 0.050 (four percent) in the reading of the optical density. Thus a cumulative error of five percent is inherent in this procedure or the modification investigated of Woelfel's method *(13)*.

A comparison of the analysis made with the two semimicroquantitative procedures outlined is presented in Table I. Untreated, soft, 12/4 cotton sewing thread was treated experimentally with a formulation of solubilized copper 8-quinolinolate (10.0 percent) extended in isopropyl alcohol. The concentration of the copper 8-quinolinolate in the bath was adjusted to a 52 percent wet pickup of the alcohol by a weighed amount of untreated thread. A skein weighing 3.30051 gm was soaked in the bath solution and then passed through the rollers as a single thread. The treated thread was air-dried before being sampled for analysis. A random amount of thread was discarded and two adjacent inches were each weighed for analysis by each of the two semimicro methods. Again a random amount was discarded and two adjacent inches taken for analysis. A total of nine samples were analyzed by each of these two methods.

It is apparent that the two methods give similar results. The correlation coefficient, r, was calculated to be +0.932 for the two methods. The adaptation of the Rose et al method *(11)* was simpler and more rapid and therefore preferred over the modification of Woelfel's method *(13)*. All further semimicroquantitative analyses were made by the method adapted from that of Rose et al.

Using samples taken from the experimentally mildewproofed thread, a comparison was made of the method reported by Rose et al *(11)* for relatively large samples and the semimicroquantitative adaptation thereof outlined herein. The results are presented in Table II. In this instance approximately seven feet were taken from the skein, weighed and analyzed by the method of Rose et al *(11)*. A random length of thread was discarded and a one-inch sample was taken for semimicroquantitative analysis. A total of four large samples (seven feet each) and four one-inch samples were analyzed. In addition, similar-size samples were removed from untreated, soft, 12/4 thread to serve as blanks.

It is apparent in these data that similar concentrations of copper 8-quinolinolate were analyzed by the method of Rose et al *(11)* and the semimicro adaptation thereof in the experimentally treated thread described above. No significant differences could be established by analysis of variance (Table II) for data obtained with large and semimicro determinations. Further, no difference was computed between the samples analyzed regardless of the method of analysis. This agreement in the amount of copper 8-quinolinolate determined in experimentally treated thread was not obtained with commercially mildewproofed thread.

ANALYSIS OF COPPER 8-QUINOLINOLATE APPLIED COMMERCIALLY TO 12 4 COTTON SEWING THREAD——A spool of commercially mildewproofed thread fulfilling military specification requirements was sampled in the same manner as was accomplished for the experimentally treated thread. In addition, eight semimicro samples were taken at random from the interior of this spool of thread. The determinations are reported in Table III.

The large sample determinations made by the method of Rose et al *(11)* are in excellent agreement. Semimicro analyses of one-inch samples gave an average concentration like that determined by the Rose et al method, but the standard devia-

TABLE I

Semimicroquantitative determinations by methods adapted from Woelfel and Rose et al of copper 8-quinolinolate applied experimentally to 12/4 cotton sewing thread

	Modification of Woelfel's method (11)			*Adaptation of method of Rose et al (9)*		
Sample no.	*Weight (mg)*	*Optical density*	*Percent Cu-8*	*Weight (mg)*	*Optical density*	*Percent Cu-8*
1	4.52	.071	1.11	4.16	.040	0.96
2	4.74	.075	1.12	4.03	.041	1.02
3	4.82	.080	1.17	3.10	.032	1.03
4	4.20	.047	0.82	4.02	.033	0.82
5	4.12	.051	0.89	4.36	.039	0.89
6	4.36	.067	1.09	4.46	.041	0.92
7	4.00	.054	0.96	4.24	.037	0.87
8	4.06	.043	0.78	4.16	.033	0.79
9	4.56	.061	0.96	4.28	.040	0.93
Average			0.99 ± .173			0.91 ± .082

Coefficient of linear correlation, r_{xy}, for analyses by the two semimicroquantitative methods computed as:

$$r_{xy} = \frac{N\Sigma xy - \Sigma x \Sigma y}{\sqrt{[N\Sigma x^2 - (\Sigma x)^2][N\Sigma y^2 - (\Sigma y)^2]}} = +0.932$$

TABLE II

Comparison of analysis of copper 8-quinolinolate in experimentally treated and untreated 12/4 thread by the method of Rose et al *(11)* and the semi-micro adaptation thereof

	Sample	*Method of Rose et al*		*Semimicro adaptation*	
		Weight (gm)	*Cu-8 (%)*	*Weight (gm)*	*Cu-8 (%)*
1	treated	.30096	.97	.00460	1.02
	untreated	.30255	0	.00429	0
2	treated	.32458	.95	.00405	.86
	untreated	.30034	0	.00397	0
3	treated	.30142	1.01	.00450	1.18
	untreated	.29869	0	.00454	0
4	treated	.31166	.98	.00446	.92
	untreated	.33415	0	.00397	0

Analysis of Variance

Source of variance	*Degrees freedom*	*sums of squares*	*Mean variance*	*F*
Between methods (method of Rose et al X semimicro)	1	.0006	.0006	<1
Between samples (treated)	3	.0397	.0132	1.91
Error	3	.0209	.0069	
Total				

F required for significance at five percent level for three degrees freedom for error and three degrees freedom for lesser mean square, 9.28. Computed F for "between samples", therefore, is not significant. Computed F "between methods" calculated to be less than one; therefore, not significant.

TABLE III

Percent copper 8-quinolinolate in commercially processed mildew resistant thread analyzed by method of Rose et al *(11)* and the semimicro adaptation thereof

	Thread from surface of spool		*Thread from interior of spool*
	Method of Rose et al	*Semimicro adaptation*	*Semimicro adaptation*
	1.01	1.18	1.02
	.95	.86	.92
	.93	1.03	.82
	.94	.84	.82
	.95	.79	.89
	.98	.93	.80
	.97	.87	1.02
	.95	.96	.70
Average	.92	.93	.97
s	± .05	± .12	± .11
2s params	.82 to 1.02	.69 to 1.17	.65 to 1.09

TABLE IV

Allocation of 12 subpieces obtained from each of six one-inch samples of thread to comprise a composite sampling

Numbered one-inch pieces from a nine - inch length of thread	Composite samples designated as numbers 4	5	6	7	8	9
	Numbered subpieces per inch of thread					
2	1,7	2,8	3,9	4,10	5,11	6,12
3	same					
4	same					
6	same					
7	same					
8	same					
Total number subpieces for composite sample	12	12	12	12	12	12

tion for any one analysis (s) was increased by a factor of 2.4. The parameters for concentrations analyzed based on a 95 percent probability (2s) were extended significantly by the semimicro method. The randomly selected one-inch samples from the interior of the spool gave results like those determined for the other one-inch samples. The variability in amount of copper 8-quinolinolate as established by semimicroquantitative analysis is thus quite apparent.

FURTHER STUDY OF THE UNIFORMITY OF DEPOSITION OF COPPER 8-QUINOLINOLATE APPLIED TO 12/4, GLAZED, COTTON SEWING THREAD——A sample of Type IB1 cotton sewing thread per Federal Specification V-T-276, "Thread, Cotton," 29 January 1958, mildewproofed commercially in accord with Military Specification MIL-T-3530, evidenced light and dark areas upon visual examination. This was suspected to be due to varied amounts of copper 8-quinolinolate. Two nine-inch lengths, one uniform in appearance and the other not, were investigated. Each of these lengths represented a single wind of the thread around the spool. The two nine-inch lengths of thread were cut into nine one-inch pieces each, the position of the pieces in each thread being preserved. The first, fifth, and ninth one-inch pieces of both threads were weighed and analyzed as discrete inches. The remaining six one-inch pieces of each thread were each subdivided into 12 pieces, the position of each subpiece being preserved. Subpieces 1 through 6 and 7 through 12 were taken successively from each of the 6 pieces so sampled to comprise samples 4, 5, 6, 7, 8, 9 (Table IV). The composite samples each containing 12 subpieces were then weighed and analyzed. In this manner the first and last inches proximal in their position on the spool and the middle inch opposite to these in position were analyzed. The remaining six samples as achieved averaged any variation that may have occurred between the first three samples. The results are summarized in Table V.

TABLE V

Percent copper 8-quinolinolate analyzed semimicroquantitatively (adaption of procedure of Rose et al) contained in commercially treated 12/4 glazed, cotton sewing thread

Appearance of thread uniform		Appearance of thread non-uniform	
Discrete inch	Composite inch	Discrete inch	Composite inch
.801	.793	2.217	.947
.800	.818	0.844	.855
.833	.754	1.016	.876
	.750		.974
	.797		.929
	.768		.847
Av .811	.780	1.359	.905
±	±	±	±
σ[1] .011	.013	.431	.022
$2S^2$[2] .734 to .846		.766 to 1.346	

[1] $\sigma = \frac{s}{\sqrt{N}}$ in which $s = \sqrt{\frac{\Sigma d^2}{N-1}}$, the square root of the sums of the deviations squared divided by one less than the number of analyses.

[2] Parameters of two standard deviations of concentrations that may be determined within 95 percent probability limits.

It is evident from these data that discrete one-inch pieces of uniform-appearing thread are in good agreement with the composite samples analyzed semimicroquantitatively from the same length of thread. Good agreement was obtained among the composite samples made from the nonuniform thread, but highly varied concentrations were determined for the discrete pieces from this length of thread.

These data demonstrate by semimicroquantitative analysis the variation in amount of fungicide actually applied. The analysis of the composite samples either for the uniform or non-uniform thread gave average concentrations lower than those of the discrete one-inch samples. Thus, randomizing extremely small portions of materials to comprise a sample for semimicro analysis results in an "averaging" of the analysis in a manner similar to analysis of large samples of material. In either case variation in amount of fungicide deposited cannot be revealed by analysis.

Figure 3
Sampling of test fabric strip (one inch by six inches) for semimicroquantitative analysis after measure of its breaking strength

RELATIONSHIP OF BREAKING STRENGTH AFTER SOIL BURIAL AND ANALYSES OF COPPER 8-QUINOLINOLATE OF THE SAME SAMPLES——Cotton duck (8.0-oz) treated to contain .554 percent copper 8-quinolinolate was sampled and ravelled to one-by-six-inch strips with the long direction parallel to the warp. The fabrics were buried in soil (per method 5762, CCC-T-191b, Federal Textile Test Methods) and, after given periods, were removed, washed, conditioned and their breaking strengths measured. Analysis of copper 8-quinolinolate [method of Rose et al (*11*)] were made on samples weighing approximately one gm. Thus the breaking strength was measured and the concentration of copper 8-quinolinolate determined for each given test fabric strip. The results are summarized in Table VI.

Semimicroquantitative analyses were made of eight samples removed from another single test fabric strip of known breaking strength. The pattern for sampling of the material for

these analyses is shown in Fig 3. Thus four analyses (samples numbered 5, 6, 7, 8) were made at the region of break and four analyses (samples 1, 2, 3, 4) were made away from the point of break. The results of these analyses are summarized in Table VII.

These data show that all semimicroquantitative analyses and all analyses of large samples were highly correlated, r = +.880. The correlation for breaking strength and large sample analyses was relatively poor, r = +.550. Equally so were correlations of semimicro analyses away from region of break and all semimicro analyses each related to breaking strength (r = +.575 and +.526 respectively). A very high correlation was obtained for semimicro analyses at region of break and the relevant breaking strength, r = +.886. Although this evaluation suggests a high correlation between semimicro analyses at region of break and the breaking strength, the possibility remains that the analyses of the "at break" samples might afford less variation than the dispersed samplings for "away from break" analyses.

Despite occasional instances in which reduced concentrations of copper 8-quinolinolate and low values of breaking strength are followed by higher analyses and greater breaking strength, the trend for chemical analyses by the semimicro adaptation, breaking strength and time in soil burial is sufficiently consistent to give the high correlations discussed previously. It is evident (Table VII) that a remarkably high correlation (r = +.886) was obtained between the semimicro analyses at point of break and the breaking strength of the same test fabric strip. Large sample analysis and breaking strength correlated as r = +.550 (Table VI).

The dependence of breaking strength on fungicide concentration is made all the more evident by analysis of variance of the semimicro chemical determinations with respect to their positions in the fabric, at or away from the area of break. Computations made for this statistical evaluation are shown in Table VIII.

The reason for the high level of significance (F = 13.93) for "between days" is immediately understandable by virtue of the magnitude of breaking strength change from the unburied fabrics and that of the test fabrics after 70 days in burial. The variance among all semimicro analyses made from one test fabric strip approaches but does not attain a significant F ratio at the five-percent level. The difference between analyses made at position 1, 2, 3, 4 (away from point of break) and 5, 6, 7, 8 (at point of break) is highly significant. The correlation (r = +.886) determined between analyses at point of break and breaking strength is therefore confirmed by the high F value (80.37) computed for these data. When analyses away from point of break are included with those made at point of break the variance is reduced to insignificance. In this latter instance differences in amount of copper 8-quinolinolate deposited even on this small area of cloth are sufficiently great, apparently, to obscure any significant trend relating loss in concentration and reduction in breaking strength. The high and low correlations obtained correspond with computed F ratios. Hence, uneven deposition of copper 8-quinolinolate is effectively demonstrated by the semimicro adaptation of the method of Rose et al *(11)*. Method of analysis and size of the sample, particularly, are important factors in evaluating deterioration of mildewproofed materials; and uneven deposition of

TABLE VI

Large sample analysis of copper 8-quinolinolate[1] applied to 8.0-oz cotton duck, the analyses made from samples of known breaking strength after given number of days in soil burial

Rose et al method of analysis of copper 8-quinolinolate in three test fabric samples

Days buried	Fabric 1		Fabric 2		Fabric 3	
	Breaking strength (psi)	Copper 8-quinolinolate (%)	Breaking strength (psi)	Copper 8-quinolinolate (%)	Breaking strength (psi)	Copper 8-quinolinolate (%)
0	125	.563	137	.514	141	.513
7	128	.638	126	.665	130	.598
14	142	.654	137	.581	130	.596
35	123	.566	153	.516	121	.497
42	135	.355	117	.260	111	.303
49	98	.289	74	.343	92	.398
56	115	.618	133	.500	127	.539
63	108	.376	78	.288	110	.377
70	109	.197	130	.239	110	.162

r_{xy} (see Table II) = +.550 for pounds breaking strength × macroquantitative anlysis.
[1] Method of Rose et al *(11)*.

TABLE VII

Semimicroquantitative determination of copper 8-quinolinolate applied to 8.0-oz cotton duck, the analyses made from samples of known breaking strength after specific number of days in soil burial

Days buried	Semimicroquantitative analysis as percent copper 8-quinolinolate			Breaking strength of relevant sample (psi)
	at point of break[1]	Away from point of break[1]	All[2] Analyses	
0	.478	.544	.511	112 ± 9.4
7	.473	.472	.472	137 ± 7.2
14	.463	.572	.446	103 ± 12.6
35	.437	.445	.441	147 ± 10.7
42	.312	.283	.297	132 ± 9.8
49	.237	.374	.305	104 ± 15.1
56	.321	.491	.406	114 ± 13.0
63	.446	.409	.428	121 ± 14.3
70	.137	.232	.184	84 ± 23.4

[1] average of 4 determinations.
[2] average of 8 determinations.

Correlation r_{xy} (See Table II)

Factors Correlated	r
Semimicro analysis × breaking strength at point of break	+.886
Semimicro analysis × breaking strength away from point of break	+.575
All semimicro analyses × breaking strength	+.526
All semimicro analyses × all macroanalyses (cf Table VI)	+.880

TABLE VIII

Analysis of variance of data obtained by semimicroquantitative determination of copper 8-quinolinolate in 8.0-oz cotton duck

Source of variance	Degrees freedom	Sums of squares	Mean variance	F
Between days	8	.782608	.097826	13.93
Among the eight samples from one test fabric strip	7	.101771	.014539	2.07
Between positions 1,2,3,4, × 5,6,7,8	1	.056428	.056428	80.37
Error	55	.386181	.007021	
Total	71	1.326988		

F required for significance at five-percent level with 55 degrees freedom for error and
8 degrees freedom for lesser mean square = 2.11
7 degrees freedom for lesser mean square = 2.18
1 degree freedom for lesser mean square = 4.02

the fungicide on a cloth could be an important variable suggesting a source of poor agreement between biological and chemical evaluations in one laboratory or between laboratories.

SUMMARY AND CONCLUSIONS

A semimicroquantitative chemical analysis for copper 8-quinolinolate applied to cellulosic materials correlated well with the procedure for relatively large sample determinations from which it was adapted. This semimicro method adapted from a spectrophotometric procedure was found to be feasible and reproducible. The variation in deposition of copper 8-quinolinolate was demonstrated by analyses made of cotton sewing thread mildewproofed with this fungicide. The correlation of breaking strength of copper 8-quinolinolate-treated 8.0 oz cotton duck following soil burial and eight semimicroquantitatively determined concentrations of this fungicide made from the same test fabric strip was computed as $r = +.886$. The correlation of analyses made away from point of break and breaking strength was determined to be $r = +.575$. Large sample analysis of similar test fabric strips gave an r value of +.550 indicating a poor correlation between breaking strength and concentration as determined. All eight semimicroquantitatively determined concentrations for a given test fabric strip regardless of their position (at or away from region of break) correlated with breaking strength as $r = +.526$. The averaging of semimicro determinations results in as poor a correlation with breaking strength as occurs when large sample analyses are correlated with breaking strength. Variations in deposition of copper 8-quinolinolate on thread and cloth does occur and is amply demonstrated by the semimicro adaptation of the method of Rose et al. Unevenness of deposition of the mildew inhibitor could be an important variable in itself and may be a source of poor agreement between successive evaluations in one laboratory or between different laboratories.

LITERATURE CITED

(1) Center, E J, and McIntosh, R M, *Ind Eng Chem Anal Ed* **17**, 239 (1945).
(2) Darbey, A, *Am Dyestuff Reptr* **42**, No. 15, 453 (1953).
(3) Delavault, R, and Irish, R, *Compt Rend* **233**, 1614 (1951).
(4) Fischer, H, and Leopoldi, G, *Wiss Veroffentlich Siemens-Konzern* **12**, 52 (1933).
(5) Haddock, L A, and Evers, N, *Analyst* **57**, 495 (1932).
(6) Moeller, T, *Ind Eng Chem Anal Ed* **15**, No. 5, 346 (1943).
(7) Muller, R H, *Mikrochem Acta* **28**, 209 (1940).
(8) Numata, I, *J Biochem (Japan)* **30**, 395 (1939).
(9) Polester, M, *Chem Listy* **43**, 228 (1949).
(10) Ringborn, A, and Sundbran, F, *Firsha Kemitramfundets Medd* **51**, 42 (1942).
(11) Rose, A, Hutchison, A Witt, Hayes, J R, and Sharkey, I R, *Am Dyestuff Reptr* **45**, 362 (1956).
(12) Stearns, E I, *Ind Eng Chem Anal Ed* **14**, 568 (1942).
(13) Woelfel, W C, *Anal Chem* **20**, 723 (1948).

• PATENT DIGEST •

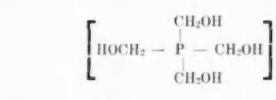
PAUL WENGRAF

FLAMEPROOFING OF TEXTILES———Phosphorus-containing amino resins G.2,03

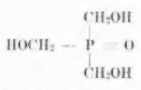
USPat 2,809,941 (Secy Agriculture———Reeves, Guthrie———Oct 15, 1957)

Condensates useful as flameproofing agents are, according to the current patent, cross-linked, hydroxyalkylated phosphorus and nitrogen-containing polymers in which each of the recurrent units contains a P-atom linked to at least two trivalent N-atoms.

The phosphorus compounds mentioned in this specification are either tetrakis - (hydroxymethyl) phosphonium chloride of the formula



subsequently termed as "THPC" or tris (hydroxymethyl) phosphine oxide of formula



termed as "THPO".

The nitrogen compounds may be amino products, such as urea or melamine, or derivatives thereof, eg, methylol urea or methylolmelamine, aliphatic amines (ethylamine, ethylene diamine), amides (formamide, acrylamide), or amino acids (glycine, lysine, etc). Thus the polymer can be formed by reacting THPC or THPO either with one of the above amino products or with the amino products *plus* formaldehyde. In the latter case, the P-compound can be incompletely reacted with the nitrogen base prior to reaction with formaldehyde, or the nitrogen base can be incompletely reacted with formaldehyde prior to the condensation with THPC (THPO), or, finally, the three compounds can be reacted simultaneously.

Among the nitrogen compounds, melamine, urea or their methylol derivatives are preferred. Whether the N-compound is present as a base linked by $-CH_2-$ to the P compounds, or as a methylolated compound linked with the $-CH_2-O-CH_2-$ group, condensation is carried out by mixing the components, applying the aqueous solution to textiles, squeezing, drying and curing.

Examples: *For a nonmethylolated N-compound*: THPC and melamine were mixed in aqueous medium, agitated for 20 minutes at 80-90° C until clearly dissolved and neutralized. The solution polymerized almost instantly and, after curing at 110° C, gave an imcombustible thermosetting resin.

For a methylolated compound: A mercerized cotton cloth was padded in a solution of THPC and a water-soluble mixture of partially reacted melamine and formaldehyde. The impregnated cloth was dried at 55-65° C and cured for four minutes at 150° C. After being washed in hot tap water, the fabric was found to be flame-resistant without being stiff.

Similar condensates have been advocated for the flameproofing of amino-alkylated cellulose in the same inventors' USPat 2,668,096 (see references below).

References cited by the Patent Office:

USPat 2,606,902 (Am Cyanamid/1952) describes ethylene amino de-

(concluded on page 614)

EFFECT OF COMMERCIAL LAUNDERING ON CEU-TREATED COTTON FABRICS

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New Orleans, La

An untreated control, cloth finished with CEU, and with a CEU-acrylate-silicone formulation were subjected to commercial laundering at four locations to determine the effect of this type of laundering on resin-treated cotton fabrics.

Soiling of the sample treated with the CEU-acrylate-silicone resin was observed as a result of laundering with dirty clothing. This appears to be due to a tendency of this finish to attract and scavenge dirt from the laundry water.

Apparently, the chlorine bleach used by these laundries did not harm the finish. The durability of the physical properties—breaking strength, tearing strength, flex abrasion, wrinkle recovery, and nitrogen content—was very good to laundering in which the souring step was well controlled. With over-souring, resin was removed by hydrolysis, resulting in decreased wrinkle recovery and an increase in most of the other properties. Investigation of the conditions of souring indicated the rate of hydrolysis to be greatly increased by elevated temperatures and by pH below 4.

INTRODUCTION

IN A previous paper the importance of resin treatment of cotton cloth for wash-and-wear and durably creased garments was described (*1*). Included in this study was a report on the excellent durability of the resistance of a creasing finish containing dimethylol cyclic ethyleneurea, a silicone, and an acrylate polymer, to laundering in the absence of chlorine bleach. In a subsequent investigation (*2*), the effect of active chlorine bleach on this finish as well as on fabrics containing only dimethylol cyclic ethyleneurea, or more correctly 1,3-bis(hydroxymethyl)-2-imidazolidinone (CEU), has been investigated by a home-type laundering process. Durability on both type finishes was excellent.

Athough durability toward home-type laundering was demonstrated, the conditions used were not comparable to those prevailing in commercial laundering. Therefore, in continuing the research mentioned above, an investigation was made under actual commercial laundering conditions, of the laundering qualities of cotton treated with the CEU-acrylate-silicone durable creasing formulation as well as with the CEU wrinkle-resistant finish without additives. Special attention was given to the effect of differences in the two laundering methods. These include, for the commercial procedure, much higher temperatures, lower ratio of water to cloth, and an acid sour treatment.

There is an impression among some textile chemists that resin-treated cloth loses whiteness on laundering due to an excessive tendency toward wet soiling. Therefore, an investigation was made to determine the wet soiling properties of the fabric when washed with dirty clothes. No tendency toward wet soiling was noticed in the previous work under home laundering conditions (*2*).

It is the object of the present work to describe the effect of commercial type laundering on cotton fabrics treated with a CEU formulation with particular attention to durability, wet soiling and damage due to chlorine bleaching or acid sour.

MATERIALS AND METHODS

The fabrics used were 80 x 80 white cotton print cloth, 3.2 oz/sq yd, which had been boiled, bleached, and desized prior to resin treatment. Three types of samples were tested: a) the untreated control; b) cloth impregnated with a solution of 7% CEU plus 1.5% catalyst (a modified zinc complex) dried at 60°C for seven minutes and cured at 160°C for four minutes in a mechanical convection oven, and process washed, with a final add-on of 3.5%; c) cloth treated with an emulsion of the type used commercially for treating yard goods and experimentally in the durable creasing of cotton garments (*1, 3, 4*). This "standard creasing formulation" consisted of 14 g of a 50% solids solution of CEU; five g of a 35% solids solution of a thermoplastic acrylate polymer; 1.5 g of a 35% solids solution of a modified, zinc salt catalyst; two g of a 40% solids solution of an emulsified silicone fluid; 0.4 g of a silicone polymerization catalyst; 0.1 g alkylarylpolyether alcohol (a softening agent); and 77 g of water. The emulsion-impregnated fabric was cured from the wet state with an electric hand iron set at 200°C. This type of treatment gives an excellent cure (*1*).

Launderings were performed at three commercial laundries in New Orleans, Louisiana, and at the American Institute of Laundering, Joliet, Illinois. Samples were laundered up to 12 times in a standard laundering procedure. Briefly, the AIL laundering procedure was as shown in Table I (*5*).

TABLE I

Operation	Time (min)	Temp (°F)	Solution
1 Suds	6	130	Built soap and alkali
2 Suds	6	160	Built soap
3 Suds	6	160	Built soap
4 Bleach	8	150	Bleach solution
5 Rinse	2	160	Water
6 Rinse	2	160	Water
7 Rinse	2	160	Water
8 Rinse	2	130	Water
9 Blue	3	100	Bluing solution
10 Sour	3	100	Sour to pH 5.0

The procedure followed by the New Orleans laundries was essentially the same as that shown in Table I except that the bleach was included in the third sudsing, and at one of the plants a temperature of 180°F was used in the second and third sudsing and in the first two rinses. Samples were laundered with batches of white shirts. Souring pH was maintained between 5.5-6, 3.8-4.1, and 3.5-3.6.

Physical testing was performed at standard conditions of 70°F and 65% relative humidity by the standard methods of ASTM with breaking strength determined on strips raveled to 80 warp threads (*6a*), flex abrasion by the Stoll method (*6b*), and tearing strength by the Elmendorf method (*6d*). Wrinkle recovery angles were measured by the Monsanto method (*6c*). Soiling was estimated by determining reflectances with a Hunter Reflectometer.

DISCUSSION OF RESULTS

WET SOILING OF SAMPLES DURING LAUNDERING —— After a single laundering, at the plant, using the highest wash temperature, the control and fabrics treated with CEU were practically unchanged. They were as white as the usual bleached fabric. However, the fabrics treated with the CEU-acrylate-silicone emulsion were gray and

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decidedly dirty. They appeared to attract and scavenge soil from the laundry water which had a large amount of soil present due to the high cloth to water ratio. These samples became progressively dirtier through the third cycle and then improved slightly. At the end of 12 cycles, the control and CEU samples were essentially indentical, with approximately four to five percent drop in reflectance, while the fabrics treated with the standard emulsion had about 20% loss as compared with the original. These results are shown in Figure 1.

It was felt that this tendency toward wet soiling is a serious fault of this finish. Investigations have been initiated to determine methods for soiling treated samples in the laboratory, the causes of soiling, and the development of formulations to avoid soiling. The results of the first of these studies have been reported (7).

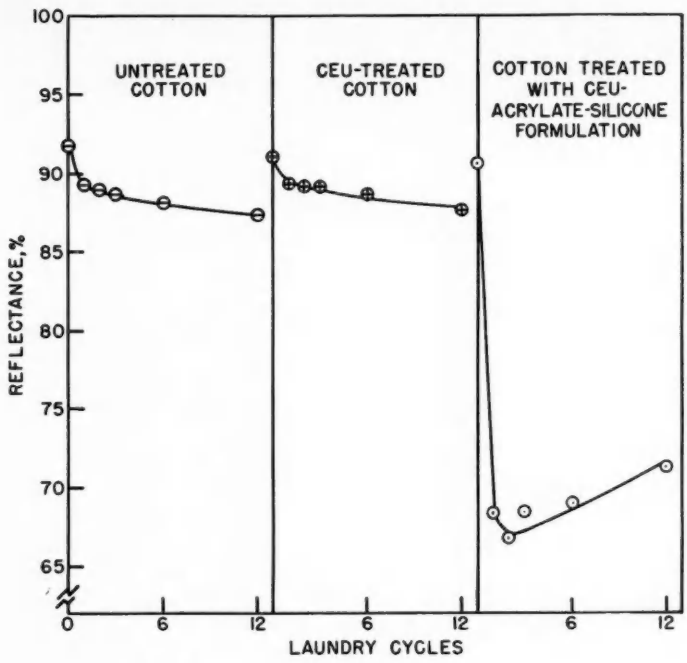


Figure 1
Wet soiling of resin treated samples during laundering

CHANGE IN TEXTILE PROPERTIES OF LAUNDERED SAMPLES ——Large losses in crease angle, abrasion resistance, breaking strength, tearing strength and resin content have been attributed to the action of chlorine bleaches on resin-treated cotton fabric (8). In a preliminary study, considerable variation in results was obtained from samples laundered at two plants.

At one, the physical properties of all test swatches showed excellent durability through 12 laundering cycles. The pertinent data on these samples are presented in Table II. It is evident that there is essentially no diminution in breaking strength and no significant change in flex abrasion. Furthermore, the wrinkle recovery values of the CEU-treated sample are unaffected by the laundering. While the values of the emulsion-treated sample have decreased somewhat, the final value of 140° (warp) after 12 launderings is still excellent.

That the resin has been retained is evident from the nitrogen content of the samples. It was found that nitrogen, which is proportional to resin content, remained approximately unchanged within the limits of experimental error on both of the treated samples. In all probability the position and extent of bonding of the resin within the cotton must be essentially unchanged throughout the laundering. Any change in this respect would result in a decrease in wrinkle recovery.

It was concluded that 12 launderings in the presence of bleach, under the conditions specified, did not damage cottons treated with CEU-resins.

At the second laundry, a very different picture of durability due to laundering was obtained. Briefly summarized, breaking strength, tearing strength and flex abrasion of the resin-treated samples increased while wrinkle recovery dropped considerably. These results point to a loss of resin which was confirmed by a reduction in the nitrogen content of from 30 to 70%.

It was felt that the loss in resin was due to some difference in laundering conditions. The main differences in commercial laundering practice are the souring procedure and temperature and alkalinity of the wash. It has been shown, for example by Cooke and coworkers (9), that urea-formaldehyde and CEU resins are much more susceptible to acid hydrolysis than melamine-formaldehyde resins. The effect of acid souring under various conditions was investigated as described below.

EFFECT OF SOURING ON HYDROLYSIS OF CEU-RESIN —— Buffered solutions, in the range of pH of 3-8, were used to determine the effect of "souring" under these conditions. In addition, a solution of strong alkali, pH 14, was included to provide a greater range of data and to investigate stability to high alkalinity. For this experiment, untreated, CEU-treated and CEU-acrylate-silicone-treated samples were exposed to the buffered solutions at 104°F

TABLE II
Durability to laundering of resin-treated and control cottons at a commercial laundry

	Breaking strength warp (lb)				*Tearing strength warp (lb)*				*Flex abrasion warp (cycles)*				*Wrinkle recovery warp (degrees)*				*Nitrogen content (%)*			
Sample	*0 cyc*	*1 cyc*	*6 cyc*	*12 cyc*	*0 cyc*	*1 cyc*	*6 cyc*	*12 cyc*	*0 cyc*	*1 cyc*	*6 cyc*	*12 cyc*	*0 cyc*	*1 cyc*	*6 cyc*	*12 cyc*	*0 cyc*	*1 cyc*	*6 cyc*	*12 cyc*
Untreated	44.3	42.2	43.3	40.9	2.1	2.0	1.9	1.8	397	406	269	324	90	97	94	98	—	—	—	—
CEU-treated	27.7	26.9	26.8	27.5	1.0	1.1	1.1	1.1	47	49	40	51	153	151	150	150	1.7	1.7	1.6	1.6
CEU-acrylate silicone treated	30.6	32.1	33.9	31.8	1.4	1.4	1.4	1.3	77	97	96	180	160	151	144	140	0.9	1.0	0.9	0.9

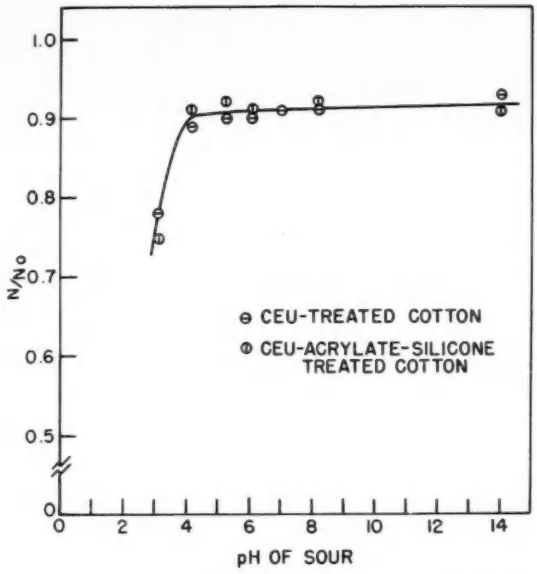


Figure 2
Stability of resin treated cottons to solutions of various pH at 104°F (40°C)

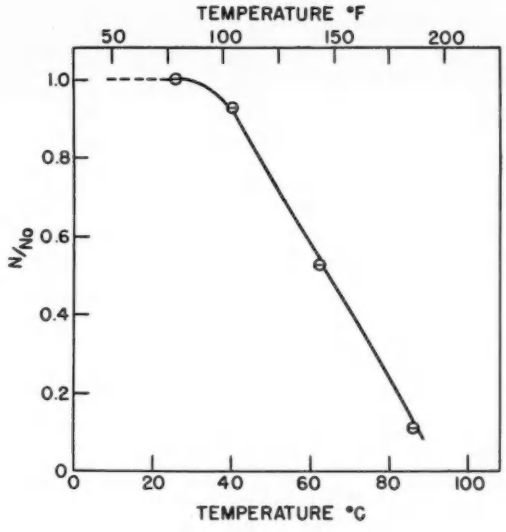


Figure 3
Loss of resin with increased temperature during souring at pH 3.6 for 30 minutes

(40°C) for 30 minutes. The time of treatment, although considerably longer than that used in actual commercial laundering, was selected to exaggerate any difference which might be attained to show the effect of pH. Figure 2 is a plot of the fraction of finish remaining after "souring", as measured by the ratio of nitrogen content after treatment to nitrogen content of the original fabric. The points for the samples treated with resin and with resin plus additives all fall on the same essentially straight line from pH 4 to 14. However, below pH 4 there is a sharp, decided break showing significantly greater hydrolysis of the resin. The fact that in the range of pH 4 to 14, all the samples are grouped at an N/N_0 ratio of about 0.9 indicates that either about 10% of the resin is not bound to the cellulose and is readily removed, although it was not removed by process wash, or that for this entire range hydrolysis of the resin proceeded to the same extent, due to 10% of the resin being bound through a labile linkage of some type. The latter seems more likely since hydrolysis of resin in water solution at higher temperature has been observed on samples of CEU-treated fabric. However, determination of the causes of this phenomenon are not within the scope of this investigation.

The textile properties of these samples were examined and found to follow the same pattern as indicated from Figure 2. In the range of pH 4-14 all samples exhibited essentially equal textile properties. Samples soured at pH 3 showed the usual effects of resin hydrolysis—increased breaking strength and decreased wrinkle recovery angle.

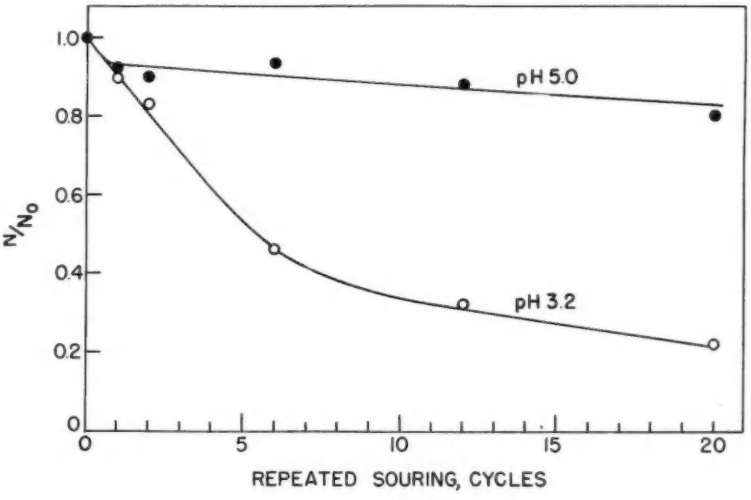


Figure 4
Comparison of the effects of repeated souring at pH 5.0 and 3.2

The rapid increase in hydrolysis with increased temperature is shown graphically in Figure 3. Here the fraction of resin remaining is plotted against the temperature of the sour solution. For this experiment, 30-minute treatments with a commercial laundry sour (*10*) were used. In the "Army Mobile Laundering Procedure" (*11*) this souring agent (sodium acid fluoride and sodium silicofluoride) is used unbuffered. The sour solution had a pH of 3.6. At 100°F, the usual souring temperature, loss of resin was about seven percent, whereas at 187°F (86°C), loss was 89%.

To further verify the injurious effects of souring, resin-treated samples were subjected to repeated souring. With sour solutions buffered to pH 5.0 and 3.2, and time and temperature set at three minutes and 104°F, respectively, samples were soured, washed with water and ironed dry. This was repeated through 20 cycles, removing samples periodically. These data are illustrated in Figure 4.

No change in properties or nitrogen content was effected by this treatment at pH 5. At pH 3.2, the value for fraction of nitrogen retained after two souring cycles is already below that for twenty cycles at pH 5. After twenty cycles at pH 3.2, 80% of the resin has been removed.

From the above it can be seen that the pH of the souring step in laundering is critical with CEU-treated fabrics. In a discussion of souring procedure (*12*), it is recommended that not less than a pH of 4.5 be used and that this be checked by the use of indicator solutions. Although it is stated that mineral acids, oxalic acid and sodium acid sulfate must be used with caution, 100% hydrofluoric acid is listed as being acceptable. It has been found that 100% hydrofluoric acid is being used by some laundries in this region. From observation and discussion of laundering practice it is apparent that occasionally the pH of souring is much lower than is safe for garments treated with CEU.

EFFECT OF ALKALINE TREATMENTS ——— A CEU-treated sample was subjected to 20 repeated seven-minute exposures at 180°F to a one percent solution of a commercial laundering alkali which was mainly sodium orthosilicate. The solution at this concentration and temperature produced a pH of 11-12. The sample was rinsed and ironed dry after each treatment. After 20 such treatments, there was no loss in resin, as measured by nitrogen content, or of wrinkle recovery. Furthermore, after the treatments, the sample retained 93% of its strength when subjected to the AATCC chlorine retention "scorch" test (*13*).

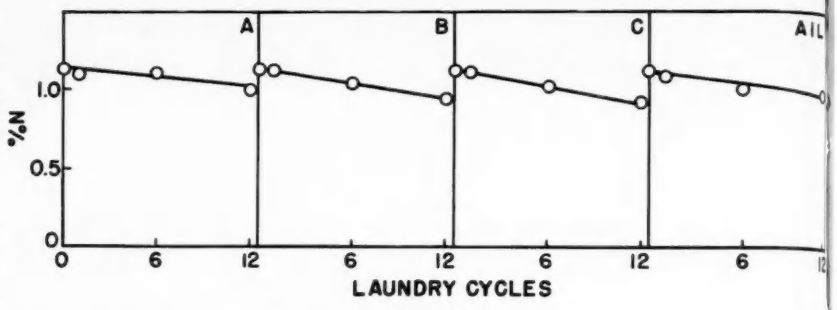


Figure 5
Commercial laundering of CEU-treated cotton at four laundry plants showing durability of finish measured by nitrogen analyses.

COMPARISON OF DURABILITY AT FOUR LAUNDERING PLANTS ——— Another set of samples was prepared and laundered in three commercial laundries and in the plant at the American Institute of Laundering. These locations were selected partially on the basis of differences in their souring procedures. The pH of the sour solutions was carefully measured and represented a range from 3.5 to 6.0.

Results of physical testing of samples laundered through 12 cycles at all four locations were similar, Table III. It is notable, however, that samples from the laundries souring at higher pH had slightly better retention of properties.

Change in resin content, as measured by nitrogen analyses, of the CEU-treated samples, is shown in Figure 5. The slightly poorer durability in this second test may possibly be attributed to lower add-ons obtained in the resin-finishing procedure. It would be expected that the lower content is closer to the threshold value necessary for excellent properties. Consequently, small losses in resin or small amounts of bond cleavage result in more pronounced changes in the physical properties. It is apparent from a study of the data on these samples that, even after the 12 cycles, wrinkle-recovery angles are still very high and in general all other properties have shown an improvement.

From the foregoing data, the authors conclude that CEU-treated cotton fabrics which are well finished with sufficient resin show excellent durability to properly controlled commercial laundering. The condition of souring at pH 5, as recommended by the AIL, should be closely observed. It is obvious that if the pH is allowed to go below 3.5, the finish may be seriously damaged.

SUMMARY

CEU-treated cotton fabrics, as well as those treated with a CEU formulation containing thermoplastic additives, used in this laboratory for "permanent creasing" studies, are remarkably stable to commercial laundering if low pH is avoided in

TABLE III
Comparison of durability to laundering of resin-treated and control cotton at three commercial laundries and at the American Institute of Laundering

Sample	pH of sour	Breaking strength warp (lb) 0 cyc	1 cyc	6 cyc	12 cyc	Tearing strength warp (lb) 0 cyc	1 cyc	6 cyc	12 cyc	Flex abrasion warp (cycles) 0 cyc	1 cyc	6 cyc	12 cyc	Wrinkle recovery W + F (degrees) 0 cyc	1 cyc	6 cyc	12 cyc
Laundry A	5.5–6.0																
Untreated		49.5	48.1	42.5	37.9	2.3	2.3	2.4	2.1	515	600	575	355	203	203	197	190
CEU-treated		25.7	32.8	28.2	30.7	1.2	1.2	1.1	1.1	135	75	55	155	309	283	294	274
CEU-acrylate-silicone-treated		37.9	32.0	32.1	32.4	1.5	1.6	1.5	1.7	210	125	180	215	304	296	314	277
Laundry B	3.8–4.1																
Untreated		49.5	46.8	44.6	39.9	2.3	2.2	1.8	1.7	515	375	300	320	203	203	207	225
CEU-treated		25.7	32.0	33.5	32.0	1.2	1.3	1.4	1.3	135	100	215	300	309	289	279	262
CEU-acrylate-silicone-treated		37.9	37.3	40.3	34.8	1.5	1.7	1.7	1.6	210	175	305	315	304	298	297	272
Laundry C	3.5–3.6																
Untreated		49.5	49.6	43.1	38.3	2.3	2.4	2.2	2.1	515	455	610	750	203	235	203	220
CEU-treated		25.7	30.1	33.7	32.4	1.2	1.2	1.4	1.4	135	205	175	265	309	266	267	266
CEU-acrylate-silicone-treated		37.9	35.0	37.3	33.5	1.5	1.6	1.8	1.8	210	250	455	350	304	315	273	268
AIL	4.7–5.4																
Untreated		49.5	42.0	42.6	39.8	2.3	2.1	1.6	1.6	515	440	355	410	203	195	195	217
CEU-treated		25.7	30.3	29.7	31.7	1.2	1.2	1.1	1.2	135	100	155	160	309	295	254	278
CEU-acrylate-silicone-treated		37.9	34.6	32.9	28.8	1.5	1.6	1.6	1.7	210	220	330	240	304	318	267	258

the souring step. This is evidenced by nitrogen analyses and by the excellent retention of the physical properties, such as breaking strength, tearing strength, flex abrasion and wrinkle recovery. At a pH below 4, as is obtained according to some souring specifications, hydrolysis of resin and loss of wrinkle resistance occur.

Some undesirable wet soiling takes place in the fabrics treated with CEU in combination with thermoplastic additives although cotton finished with CEU alone was as resistant as untreated cotton to soiling. The causes of this soiling have been described in another report.

ACKNOWLEDGMENT

The authors wish to acknowledge the cooperation of E J Favre of Chalmette Laundry, V B Bagert of Bagert's Laundry, and Henry Tung of Oriental Laundry, all of New Orleans, Louisiana, and Lee G Johnston and Norman Duckworth of the American Institute of Laundering, Joliet, Illinois, for carrying out the launderings reported in this paper. They wish also to acknowledge the aid of McLean George for textile testing, of Harold P Pastor for Kjeldahl determinations, of George I Pittman for preparing the drawings, and of Harry B Moore, Terrence W Fenner and Ronald H Sunseri for assistance in laboratory procedures.

LITERATURE CITED

(1) Reid, J D, Mazzeno Jr, L W, Reinhardt, R M, and Markezich, A R, *Textile Research J* **27**, 252 (1957).
(2) Reinhardt, R M, Mazzeno Jr, L W, Fenner, T W, and Reid, J D, *Am Dyestuff Reptr*, **47**, No. 15, 505-8 (July 28, 1958).
(3) Graham, R T, *Textile Research J* **27**, 270 (1957).
(4) Hurwitz, M D, *ibid* **27**, 267 (1957).
(5) Johnston, L G, American Institute of Laundering, Joliet, Illinois. Special Report No. 231 (1956).
(6) ASTM Designation a) D39-49; b) D1175-55T; c) D1295-53T; d) D1424-56T, American Society for Testing Materials, Philadelphia, Pa.
(7) Mazzeno Jr, L W, Kullman, R M H, Reinhardt, R M, Moore, H B, and Reid, J D, *Am Dyestuff Reptr* **47**, P299-P302 (May 5, 1958).
(8) Walmsley, L L, *ibid* **46**, P237 (1957).
(9) Cooke, T F, Roth, P B, Salsbury, J M, Switlyk, G, and van Loo, W J, *Textile Research J* **27**, 150 (1957); *Am Dyestuff Reptr* **46**, P585 (1957).
(10) U S Federal Supply Service, Fed Spec No. P-L-137, U S Govt Printing Office, Washing, DC.
(11) U S Federal Supply Service (Fed Spec CCC T-191b), "Textile Test Methods," Method 5556, U S Govt Printing Office, Washington DC.
(12) Anon, "Better Laundering," 2nd ed, p58, Procter & Gamble, Cincinnati, Ohio, 1951.
(13) American Association of Textile Chemists and Colorists, "1957 Technical Manual and Year Book," **33**, p121, Howes Publishing Co, Inc, New York, 1947.

Abstracts

Optical Brightening Agents

Adams, D A W, Dyer **119**, 897, June 20, 1958

Well over 100 British patents have been granted and 30 or more manufacturers throughout the world are producing a range of between 150 and 200 different optical brightening agents. In 1955, production in the USA was 1375 tons—double the figure three years previously. Today the production there is still higher—a sales value of $15,000,000 has been quoted—and the world production must be at least 2500 tons. Prices have dropped steadily over this period to figures of between 15s and 40s per lb, depending on strength, complexity and properties.

The author states that the biggest proportion of optical brightening agents goes into soaps and detergents, the rate of usage being between 0.05 and 0.1%, depending on the type of whitener. Since most white goods in the weekly wash are cottons, the brighteners used in soaps and detergents should have good affinity for cellulose.

Many white cotton fabrics are now treated with an optical brightener during bleaching or finishing. The essential features are reasonably good solubility and a somewhat lower substantivity than that required for use in soaps and detergents. Too high substantivity may lead to uneven application. Complications may arise if the goods are finished with resins, since many of the brighteners are not stable to acid catalysts.

Most optical brighteners used today on cellulose have at least moderate lightfastness.

The brighteners giving the best results on cellulosic materials cannot be expected to give good results on other fibers. A few brighteners are available, however, which may be used on wool, but their fastness to light is somewhat inferior. For nylon, generally the wool types are used, or special dispersed or cationic types.

It is possible to add certain optical brightening agents to viscose dope during spinning.

The author reviews the chemical constitution of the various types of brighteners, including miscellaneous types made specifically for protein fibers.

General Calendar

AMERICAN OIL CHEMISTS SOCIETY
Oct 20-22 (Sherman Hotel, Chicago); April 20-22, 1959 (Roosevelt Hotel, New Orleans, La; Sept 28-30, 1959 (Statler Hotel, Los Angeles, Calif)

AMERICAN SOCIETY FOR TESTING MATERIALS (COMMITTEE D-13)
Oct 14-17 (Sheraton McAlpin Hotel, New York, N Y)

DIVISION OF CLOTHING AND TETXILES IN EXTENSION, TEACHING AND RESEARCH, UNIVERSITY OF GEORGIA
Nov 7-8 (2nd Textile Seminar, Georgia Center for Continuing Education, Athens, Ga)

DRUG, CHEMICAL & ALLIED TRADES SECTION, NYBT
Sept 11-14 (Annual meeting, Sagamore Hotel, Folton Landing, Lk George, NY)

THE FIBER SOCIETY
Sept 9-10 (Montreal, Que); April 29-30, 1959 (Fontana Village, NC)

INSTITUTE OF TEXTILE SCIENCE
Sept 8 (Sixth Scientific Session, McGill Univ, Montreal, Que)

INTERNATIONAL TEXTILE MACHINERY EXHIBITION
Oct 15-25 (Belle Vue, Manchester, England)

NATIONAL COTTON COUNCIL OF AMERICA
Oct 1-2 (Chemical Finishing Conference, Washington, DC)

SOUTHERN TEXTILE EXPOSITION
Oct 6-10 (Greenville, SC)

SYNTHETIC ORGANIC CHEMICAL MANUFACTURERS ASSOCIATION OF THE UNITED STATES
Luncheon meeting at the Hotel Roosevelt, New York, NY; Sept 9, Oct 8, Nov 12

Patent Digest———

(concluded from page 608)

rivatives of phosphoric acid for use as textile chemicals, for producing plastics, and possibly as therapeutic agents.

USPat 2,668,096 (Secy Agric/1954): flameproofing of fabrics consisting of aminized cellulose with alkyl phosphonium halides and reaction products thereof with condensates of melamine, urea, etc, with formaldehyde.

FLAMEPROOFING OF TEXTILES———Treatment with polymers of dialkenyl-monochloromethane phosphonates G,2,03

USPat 2, 811, 469 (Victor Chemical Works——Costello———Oct 29, 1957)

According to the preamble to this patent, a simple and highly effective flameproofing along with a wash-resistant effect can be attained on fabrics by applying a single treating solution. It is reported that tensile strength of the fabrics is even increased in some instances.

This patent covers a treatment with polymers of unsaturated (alkenyl-) esters of monochloromethane phosphoric acids, particularly of diallyl- or dimethylallyl-chloromethane phosphonates. These esters may be prepared, according to USPat 2,714,100 [Victor Chem Works—Toy and Rottenbury (1952)], by reacting an unsaturated alcohol with chloromethane phosphorus oxydichloride. Upon partially polymerizing this product in the presence of a polymerization catalyst (benzoyl peroxide), precipitating the polymer by hexane, and dissolving the dried polymer freed from adherent monomer in 30/70 methanol ethylene dichloride, one obtains a solution which may be applied to the goods. After the solvent is removed and curing is done at about 100° C for one-half hour, it is claimed that a durably wash-resistant effect is obtained while the strength of the cloth is unimpaired and the hand is still satisfactory.

Example: Distilled diallyl chloromethane phosphonate was dissolved in an equal amount of Chlorex (β-β'-dichloroethylether) and heated to 100° C. Then some benzoylperoxide was added and polymerization started until the viscosity, correlated with the degree of polymerization, increased about to three times the original value. The resulting prepolymer was poured into nexane to separate the hexane-soluble monomer from the precipitated polymer. The latter was dried, powdered, and dissolved in methanolethylene dichloride mixture (see above). A piece of cloth was impregnated, squeezed and cured at 100° C. The cloth's weight increased by 26.4%; when subjected to the flame test, it had a burning time of 8.6 seconds and a narrow char length of 5¼ inches. After the specimen was washed for 15 minutes in a 0.5% soap solution at 140° F, the corresponding figures were 11 seconds and four inches char length respectively.

Among the references cited by the Patent Office:

USPat 2,425,765 (Victor Chem Wks/1947) describes diallyl-phenyl-phosphonates and other compounds of this new class as useful plasticizers and flameproofing agents.

USPat 2,574,515 (Glenn L Martin Co/1951): tetrabromo diallyl phosphonamides are claimed to be efficient flameproofing agents, superior in this respect to the corresponding chlorinated compounds.

USPat 2,601,520 (Shell Development Co/1952) refers to dialkenyl-substituted phosphonates and polymers thereof, eg, diallyl phenyl methane phosphonate, as flameproofing agents.

USPat 2,714,100 (Victor Chem Wks/1955) (see above) describes diallyl-monochloromethane phosphonate and polymers as flameproofing agents.

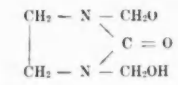
PLISSÉ EFFECTS———by applying hydroxymethylated imidazolidone and printing with shrinking agents D,1

USPat 2,810,624 (Rohm & Haas Co———Wardell ———Oct 22, 1957)

Plissé fabrics of low-count construction can be produced quite generally by locally applying shrinking agents such as thickened caustic. According to the preamble to this patent, this method *cannot be used successfully for high-count fabrics* or coarse yarn fabrics requiring high-twist yarns to produce the effects desired.

The object of the current invention is to provide dimensionally stable, improved plissé effects on all kinds of cellulosic fabrics, including tightly woven products, such as chambrays or gingham, and fabrics composed of coarse yarns, such as denims or ducks.

The patented method calls for a pretreatment by padding with a 1-3 bis (hydroxymethyl)-2-imidazolidone (N-N′ bis methylol-ethylene urea)



as a linear cross-linking agent, followed by printing with a shrinking agent, preferably a thickened caustic solution.

In contrast to precondensates that form tridimensional resins of the urea- or melamine-formaldehyde type, which modify the optical and other properties of the fibers and tend to change the hand of the fabrics, the imidazolidone derivative is a cross-linking agent, incapable of forming these kinds of tridimensional condensates. Thus the touch or the aspect of the substrate are not altered. The dyeing properties of the original material are preserved; moreover, transparentizing by the subsequently applied caustic is prevented.

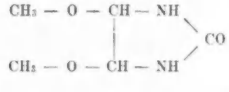
One to four percent owf of the imidazolidone derivative is applied from aqueous solution together with an acid catalyst, eg, a salt of a hydroxy aliphatic amine, and then dried and cured, whereby the fabric is stabilized against dimensional change upon washing. The second step consists of printing the caustic paste (optionally, a strong quaternary ammonium hydroxide). Preprinting resists (thermoplastic resins) and overall padding with caustic solution is within the scope of the patent. Application of a water-repellent agent (Zelane type) prior to the application of the caustic proved to produce sharper outlines of the printed effects.

Example: A cotton gingham was padded in an aqueous solution of 1.5% of the above-mentioned imidazolidone derivative, 0.3% 2-methyl-2-amino propanol hydrochloride, and a dispersion of acrylic ester polymers. It was then dried under tension and cured at 320° F for 1.5 minutes. Thereupon an aqueous paste of 35% caustic soda, thickened with polyvinylalcohol, was printed, after which the fabric was washed and neutralized with bicarbonate and washed again. The plissé effect reportedly remained unchanged even after fifteen launderings.

Among the references cited by the Patent Office:

Brit Pat 547,846 (A G Cilander/1942) mentions the decreased swelling properties of aldehyde-pretreated cellulosic fibers when exposed to the action of strong caustic.

French Pat 1,061,107 (Bad Anilin- & Sodafabrik/1954) describes a crease-resistant and stabilizing treatment with methylol compounds of the type



Proceedings of the

American Association of Textile Chemists and Colorists

Copyright, 1958, American Association of Textile Chemists and Colorists

September 8, 1958

Convention Notes

TENNYSON GUYER, whose speaking career numbers more than 8000 talks, will serve as the featured speaker at the annual banquet to be held Friday evening, October 31st, during the 37th National Convention in Chicago, Ill.

Dr Guyer has had an outstanding career in public speaking, which began in 1930, following his graduation from Findlay, Ohio, High School. Born November 29, 1912 in Findlay, the son of the late William Harris Guyer, president of Findlay College, Dr Guyer was graduated from Findlay College in 1934. He holds the following degrees: AB, MA—Litt D, DCS (Commercial Science), DD (Doctor of Divinity), and LL D (Doctor of Letters). He holds honorary doctorates from Findlay College, Tiffin Univ, and Pioneer Seminary.

Dr Guyer has served as a social worker, Federal Relief Administration; supervisor, National Youth Administration; minister, Churches of God in North America, 1935-45, Celina, O; State overseer, Findlay College, 1945-48; mayor of Celina, 1941-43; director of public relations for Cooper Tire & Rubber Co, 1951; State central committeeman for the Republican Party, 1954-56; and president of the International Platform Association, 1956-58.

Author of "Blueprints for Youth" and "Institution or Destitution", Dr

Tennyson Guyer

Guyer has delivered more than 850 Commencement addresses. His radio program on Findlay radio station WFIN has been heard by listeners in the area since 1950.

Although an unsuccessful candidate for Lt Governor of Ohio in 1956, Dr Guyer will take office in the Ohio Senate in January, having been elected from the 30th-33rd Senatorial District without opposition in the primary and general elections.

Married to Edith Mae Reuter, daughter of the village doctor, June 10, 1944, Dr Guyer has two daughters, Sharon, 13, and Rosie, 8.

Dr Guyer has traveled in 13 foreign countries in Europe, Africa and Asia.

He is a member of the Scottish Rite, Knights Templar, Elks, Exchange Club (life honorary member), Lions International, State Sheriffs Assoc, Ohio Chiefs of Police, Ohio State Highway Patrol, and the College Church of Findlay, Ohio.

Employment Register

This column is open for two insertions per year, per member, without charge. Blanks can be obtained from, and filed with, the secretary of the Association, Post Office Box 28, Lowell, Mass. It is understood that these will be open to inspection by prospective employers who can obtain further information from the secretary.

58-15

Education: BS, textile engineering, Bradford Durfee College of Technology; Norwich Univ & Univ of Massachusets (army specialized training); Boston Univ.

Experience: Dyeing and finishing, sales and sales representative, and assistant sales manager.

Position desired: Technical sales.

Location: South coast.

Age: 31; single; references.

8-25, 9-8

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AATCC Calendar

COUNCIL, COUNCIL COMMITTEES, RESEARCH COMMITTEES
Sept 17-20 (Hotel Charlotte, Charlotte, NC); Oct 30-Nov 1 (Conrad Hilton Hotel, Chicago, Ill); May 1959 (Montreal, Canada)

NATIONAL CONVENTIONS
Oct 30-Nov 1, 1958 (Hotel Conrad Hilton, Chicago); Oct 8-10, 1959 (Sheraton Park and Shoreham Hotels, Washington, DC); Oct 6-8, 1960 (Sheraton Hotel, Philadelphia, Pa); September 28-30, 1961 (Buffalo, NY); 1962 (Southern Region)

DELAWARE VALLEY SECTION
Sept 12 (Wilmington Meeting), Oct 17, Dec 5

HUDSON-MOHAWK SECTION
Oct 3 (Jack's Restaurant, Albany, NY); Nov 14 (Chelsea House, Tribes Hill, NY); Jan 16 (Jack's Restaurant, Albany, NY); Mar 27 (Chelsea House, Tribes Hill, NY); Mar 15 (Ladies night, Albany area); June 26 (Annual outing—Antlers Country Club, Tribes Hill, NY)

METROPOLITAN SECTION
Oct 3 (Kohler's Swiss Chalet, Rochelle Park, NJ)

NIAGARA FRONTIER SECTION
Sept 26 (Niagara Falls, Ont); Dec 5 (Buffalo, NY); Feb 20 (Niagara Falls, Ont); April 4 (Hamilton, Ont—joint meeting with CATCC)

NORTHERN NEW ENGLAND SECTION
Sept 12 (Outing—Wachusett Country Club, West Boylston, Mass); Oct 17 (QM Research & Development Ctr, Natick, Mass); Dec 5 (Hotel Vendome, Boston, Mass); Jan 16 (Colonial Club, Lynnfield, Mass)

PIEDMONT SECTION
Sept 19-20 (Hotel Charlotte, Charlotte, NC)

RHODE ISLAND SECTION
Sept 26 (Management Night — Warwick Country Club); Oct 23 (Providence Engineering Society); Dec 4 (Annual Meeting—Johnson's Hummocks Grille, Providence)

SOUTH CENTRAL SECTION
Sept 13 (Hotel Patten, Chatanooga, Tenn)

SOUTHEASTERN SECTION
Sept 13 (Ida Cason Callaway Gardens, Chipley, Ga); Dec 6 (Atlanta, Ga)

WESTERN NEW ENGLAND SECTION
Sept 26 (Hartford, Conn); Oct 24 (Annual Meeting—Rapp's Restaurant, Shelton, Conn; Dec 5 (Rapp's Restaurant, Shelton, Conn)

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Northern New England Section———

NYLON CARPET – GROWTH AND TECHNOLOGY*

GRAHAM M RICHARDSON and JOSEPH W LYNCH

Technical Service Section
Textile Fibers Department
E I duPont de Nemours & Co, Inc
Wilmington 98, Delaware

Richardson

Lynch

The authors emphasize the tremendous growth of the use of nylon fiber in carpeting in the last few years, and their data indicate that a tremendous upswing has occurred during the last two years. The principal reasons for the acceptance of nylon as a carpet fiber are its durability, its recovery from crushing, its price stability, its versatility (as to spinning systems and carpet styles), ease and performance of twist setting, ease of cleaning, mildew and insect resistance, nonsupport of combustion, and ease of dyeing to a fast range of shades.

The authors illustrate each of the above points and further indicate that Du Pont studies have resulted in 15-denier nylon carpet staple as the best choice at the moment. This staple is made in four types as follows: Type 100 has a bright luster; Type 101, which is "crimp set" in bright luster; Type 600, which is dull; and Type 601, which is "crimp set" in a dull luster.

Dyeing methods are discussed and a wide range of color types and dyeing methods indicated. The ability to set twist is discussed also, as well as means of making the twist setting more uniform by "dry tumbling" of the yarn skeins and before setting.

The control of fuzzing and pilling has been subject to considerable study and these problems have been well rationalized as the result of development of test methods and mill processing procedures.

Considerable testing has been done by the Du Pont Company, most of which has demonstrated that nylon makes an extremely durable carpet material. An opinion survey was conducted by a marketing research organization, the result of which indicated a very satisfactory acceptance by the consumer. 76% of the owners of nylon carpets said they would purchase that type in the future.

INTRODUCTION

MANY synthetic fiber developments have had a great impact on the textile industry. Fiber producers devote considerable research to the development of products having performance characteristics designed to meet specific consumer requirements. When this research is properly conducted, one hits "Jack and the Bean Stalk" type growth like Orlon acrylic fiber in sweaters, nylon in textured yarns, or Dacron polyester fiber in wash-and-wear garments. In looking over the current trends of synthetic fibers in the textile industry, it appears that "Use Research" has planted another fast-growing seed. We refer to the engineered development of a nylon fiber for use in carpets.

Over the past few years it has become increasingly evident that the supply of natural fibers is inadequate to meet the increasing need of the textile industry. This is certainly true of wool for carpets, and it appears that several factors contribute to the problem. There has been an increase in the number of consumers, the standard of living has been raised, part of the producing world has been cut off by Communist domination and we have been told that the sheep population has decreased in recent years. Violent price fluctuations and uncertain supplies of wool have joined with the increased requirement for floor covering to make it imperative that new, soft floor-covering materials be developed.

In considering possible fiber candidates for use in carpets, one should recognize the fiber properties which are of special interest in carpets.

These are:

1) Durability
2) Crush recovery
3) Price stability
4) Versatility
   a) Spinning system
   b) Carpet styles
5) Ease and permanence of twist setting
6) Ease of cleaning
7) Mildew and insect resistant
8) Nonsupport of combustion
9) Ease of dyeing to a fast range of shades.

Early in the development of nylon, it was apparent that it had great potential as a carpet fiber. Accordingly, a research and development team was assigned to the project of developing a suitable nylon fiber for this important use. In 1947, the first commercial nylon carpet production was started with a single manufacturer, NyeWait Carpet Company, Inc. This was a high quality, well-constructed carpet.

Today there are at least 23 manufacturers making a wide variety of carpets. The number of styles has increased from a single construction to approximately 95. This growth is shown in Table I.

TABLE I
Growth of Du Pont nylon carpet styles

Year	Number of styles	Number of manufacturers
1954	6	3
1955	9	5
1956	28	11
1957	65	18
1958	95	23

The growth of nylon in carpets, which was slow during the past ten years and even up to mid-1956, suddenly burst into overwhelming demand immediately following the June 1957 market. Figure 1 gives a graphic picture of this dynamic growth.

This curve illustrates the continued consumer acceptance of nylon in carpets.

Up to now we have discussed a few of the aspects of carpets and carpet fibers. The significance of nylon to this industry has been indicated. Now let's turn our attention to the technology of dyeing and finishing this nylon carpet fiber.

DISCUSSION

A) GENERAL DYEING CHARACTERISTICS———Nylon for carpets is being dyed commercially by raw stock, yarn, and piece-dyeing methods in a complete range of shades with good fastness. The choice of method depends on the available

* Presented March 28, 1958 before the Northern New England Section at the Hotel Commander, Cambridge, Mass.

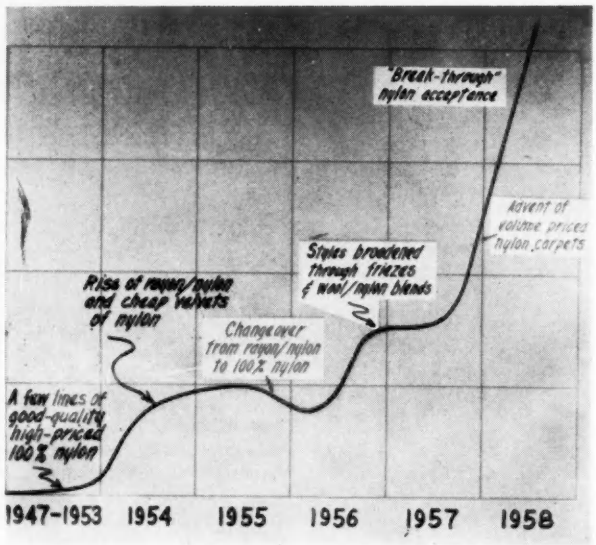


Figure 1

equipment and the type of carpet to be made. Generally, nylon is considered to be an easy fiber to dye and many dyers have reported that nylon is easier to handle in the dyehouse than are other carpet fibers. One of the contributions made to carpet by nylon is its durability to wear. Fortunately, dyes of excellent fastness are available to take advantage of the long wear life of nylon.

Before discussing nylon dyeing, it is necessary to differentiate between the various types of nylon being sold for use in carpets and their effect on dyeing and on the fastness properties of dyes. Du Pont is currently offering several types of 15-denier-per-filament nylon carpet staple and tow. They are shown in Table II.

Type 101 and Type 601 have been subjected to a crimp-setting operation. This operation has caused sufficent molecular rearrangement of the fiber to permit more rapid dyeing and deeper dyeing when dyed competitively with their companion products. Types 600 and 601 dye very much like Types 100 and 101 except that a greater quantity (approximately 40-50% more) of dye is required in the case of the dull yarns for a given shade. This does not mean that the nylon has been changed in its dyeing characteristics, but only recognizes the fact that more color is required to drown the effects of the white pigment in the fiber. Commercial interest in dull staple has been mainly in obtaining subdued luster in carpets by intimate blending with bright staple. However, the difference in apparent dye pickup between Type 600 and Type 100 can be utilized to advantage in obtaining tweed or Moresque effects.

TABLE II
Types of 15-denier nylon carpet staple

Type		*Luster*
100		bright
101	"crimp set"	bright
600		dull
601	"crimp set"	dull

Nylon has receptivity to nearly all classes of dyes. Disperse and acid dyes including neutral (metallized), acid (metallized) and chrome types have been used extensively. Disperse dyes are used for dyeing nylon carpets where maximum levelness and uniformity are required. However, they exhibit only moderately good fastness to light and washing. Because of the outstanding durability of nylon in carpets, it is recommended that dyes exhibiting maximum fastness properties, particularly lightfastness, be utilized. The neutral (metallized) dyes meet this requirement best. On the other hand, it should be recognized that these colors do not level as readily as the disperse colors.

In considering dye selection for nylon carpets, one must compare dye lightfastness for typical carpet shades. Table III shows lightfastness ratings of the same shade dyed with neutral (metallized) dyes and disperse dyes made by Du Pont.

The above values do not hold true for Type 600 nylon. In this case, the dye lightfastness of disperse colors is even poorer while the fastness of acid dyes is substantially the same.

For the purpose of this discussion, it is desirable to separate the various dyeing operations in terms of stock, tow, yarn, and piece dyeing. These will be discussed separately in the following paragraphs.

TABLE III
Lightfastness ratings of the same shade dyed with neutral premetallized and disperse dyes on 100% nylon

			Rating after 80 hours exposure in Florida	
		Material	*Sunlight*	*Under Glass*
TAN				
0.19 %	Capracyl Brown 2R			
0.015%	Capracyl Yellow NW	nylon	good	4R
0.064%	Capracyl Gray GN			
0.038%	Acetamine Yellow CG			
0.11 %	Latyl Cerise B	nylon	poor	1G
0.044%	Celanthrene Brilliant Blue FRN			
ROSE				
0.07 %	Capracyl Brown 2R			
0.06 %	Capracyl Gray GN	nylon	good	4RW
0.012%	Capracyl Yellow NW			
0.07 %	Acetamine Yellow CG			
0.19 %	Latyl Cerise B	nylon	poor	1Y
0.06 %	Celanthrene Brilliant Blue FRN			
BLUE				
0.56 %	Acid Green 25 (CI 61570)			
0.11 %	Capracyl Yellow NW	nylon	moderate	2R
0.08 %	Capracyl Brown 2R			
0.18 %	Celanthrene Brilliant Blue FRN			
0.035%	Acetamine Yellow CG	nylon	poor	1RW
0.04 %	Latyl Cerise B			
GRAY				
0.06 %	Capracyl Gray GN			
0.003%	Capracyl Yellow NW	nylon	very good	5-4W
0.005%	Acid Orange 60			
0.038%	Acetamine Yellow CG			
0.11 %	Latyl Cerise B	nylon	poor	1Y
0.05 %	Celanthrene Brilliant Blue FRN			

Ratings

Degree of change	*Direction of change*
5—none or negligible	D—duller
5-4—trace	G—greener
4—noticeable	R—redder
3—appreciable	W—weaker
2—considerable	Y—yellower
1—very much	

B) SPECIFIC DYEING METHODS——

Stock Dyeing. Traditionally, this method of dyeing has been used by the dyer supplying dyed stock for the spinning of carpet yarns on the woolen system. This offers an economical means of obtaining maximum shade uniformity when mixing large lots or blending different types or grades of fibers. Uniform lots in the magnitude of 25,000 lbs are prepared through stock dyeing and blending.

Premetalized dyes are used almost exclusively for stock dyeing. The following dyeing procedure is recommended by Du Pont:

Volume: stock ratio = 8:1.

1) Set a bath at 80° to 100°F with
   0.5 to 2.0% Duponol D Paste surface-active agent (a long-chain alcohol sulfate)
   0.5 to 2.0% Capracyl Leveling Salt (a nonionic surfactant)
   3.0 to 5.0% ammonium acetate
   X.X% dyes
2) Circulate the bath for 10 minutes.
3) Raise the temperature to the boil over a period of 45 minutes and hold for 45 minutes.
4) Exhaust the dyebath with 3.0 to 5.0% of ammonium sulfate. It is sometimes necessary to use a small amount of acetic acid to exhaust shades that contain Acid Green 25.
5) Sample, make adds if necessary, drop the bath, and rinse.

Stock dyeing removes the antistatic and lubricative finish on nylon staple. These finishes must be replaced to facilitate spinning. The following combination based on the weight of stock has been successful when exhausted onto the fiber from the final rinse: 0.75% Avitex R textile processing agent (a high alkylamine), and 0.75% Methacrol Lubricant K. These products should be added to the final rinse at 80 to 90°F and exhausted onto the fiber by raising the temperature to 120°F over a 15-minute period. The products will exhaust uniformly under these conditions at a pH of 6.0. Some mills prefer to atomize spinning lubricants on the dried staple at the pickers. If nylon is being blended with wool, the fibers should be oiled separately.

Stock dyeing tends to set the crimp in Type 100 Du Pont nylon staple so that it processes through carding and spinning similarly to crimp set 101 nylon staple and gives a similar texture in carpets. This, of course, should be proven to the user's satisfaction before production is undertaken.

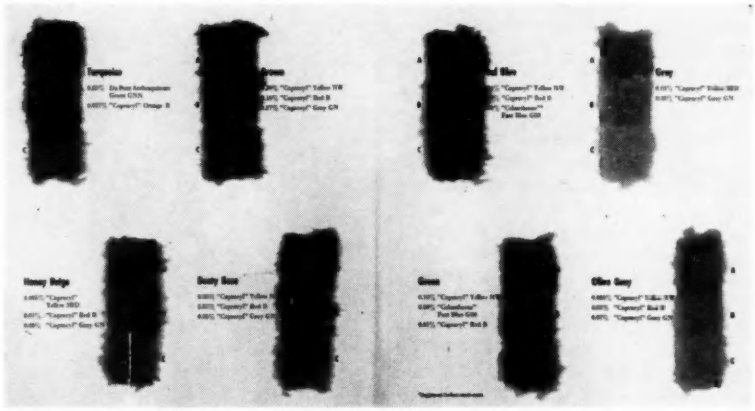


Figure 2

If temperatures above the boil are utilized in stock dyeing, one may take advantage of the little-known transfer properties of neutral (metallized) dyes at temperatures in the range of 235°F in the presence of a nonionic surfactant such as Capracyl Leveling Salt.

In Figure 2, each carpet swatch is divided into three parts, namely A, B, and C. In each case samples A and B were dyed at 235°F with the indicated formula. Transfer tests were made by placing sample B and an undyed sample in a mock dyeing bath. Sample C shows that approximately 50% of dye was transferred from sample B after 30 minutes at 235°F*(1)*.

Sufficient development data have been gathered in the laboratory to convince us that nylon stock will lend itself to continuous stock dyeing (*2*). In fact, the dye rate and uniformity of nylon indicates that it should be more readily adapted to continuous dyeing than wool. However, this remains to be proved in large-scale commercial tests. Some of the possible advantages of continuous dyeing of nylon raw stock over kettle dyeing are as follows:

1) More economical dyeing because of savings in water, steam, labor, and time.
2) Stock is subject to less packing thereby being in a better condition for the subsequent carding and spinning operations.
3) In addition, the fastness and uniformity are equivalent to kettle dyeing.

Briefly summarized, the continuous stock dyeing methods involve:

1) The use of a continuous stock scouring train with the initial bowls being utilized for dyeing. Nylon does not require a prescour so the dry stock is fed into the first bowl containing the dye solution under conditions where dyeing will occur in 60 to 90 seconds. The dyebath would contain a nonionic surfactant, neutral (metallized) dyes, and formic acid. The bath is maintained at a pH between 3.5 and 4.0 and a temperature above 205°F. Continuous circulation is necessary.
2) Intermediate bowls may be used for rinsing; however, laboratory work indicates this step is not necessary.
3) Finish may be applied in the final bowl. Here a substantive finish, such as a combination of Avitex R and Methacrol Lubricant K, is suggested.
4) Stock would then pass through the continuous dryer to storage or blending.

Another continuous dyeing method for nylon carpet staple under laboratory development is that carried out in the Du Pont Flowstock Process (*3*). This is accomplished by feeding dry nylon staple into a closed system containing a continuously flowing dye solution maintained at dyeing conditions similar to those used in the continuous bowl method. Dyed nylon stock emerging from the effluent end of the dyeing "pipe" can be extracted by squeezing and finish may be continuously applied by spray methods.

Yarn Dyeing. Yarn dyeing of nylon for use in carpets or rugs is generally practiced where a need exists for small lots, custom colors, tweed or moresque effects, or a special textured appearance.

Yarn dyeing of nylon is generally done in skein form and is handled in both raw stock kettles and skein-dyeing equipment, Neutral (metal-

lized) dyes are generally used for nylon yarn when the pack method is used. However, in some instances where the circulation characteristics of the equipment are not favorable or where the yarn twist is very hard, it is necessary to use the better leveling disperse colors. Nylon yarns used in frieze or nubby textured carpets require twist setting, and this may be accomplished simultaneously with yarn dyeing *(4)*. For best twist retention, temperatures in the range of 235°F are suggested. This requires the use of a stock-dyeing kettle capable of being subjected to pressure conditions. It should be noted that the same conditions are conducive to improved leveling of neutral (metallized) dyes.

A typical procedure for simultaneous dyeing and twist setting nylon frieze yarn is as follows:

1) Treat the dry skeins in a rotary tumbling device without heat for about five minutes in order to develop the maximum and uniform kink effect.
2) Load the relaxed skeins evenly into the stock dyeing kettle. Wet out with water containing 1.0% Capracyl Leveling Salt and circulate water for 10 minutes at 180°F. Lower the temperature to 100°F by flood rinsing. Add in the order indicated (percentages are based on weight of goods):
   4 oz antifoam agent (per 250 gallons)
   0.5% ammonia
   2.0% Capracyl Leveling Salt
   2.0% Duponol D Paste surface-active agent.

The combination of Duponol D Paste and Capracyl Leveling Salt is especially effective for good penetration and leveling of high twist yarns. The dyes are dissolved and added to the machine and the temperature is raised to 200°F over a period of 45 minutes and held for one hour. If necessary to obtain exhaust of the dye, acetic acid may be added slowly over the next one-half hour. The temperature is then raised to 235°F and held for one-half to one hour. The yarn is then cooled, rinsed, and extracted. If possible, the use of an antistatic or lubricant finish should be omitted.

Another important aspect of yarn dyeing is that of dyeing blends of nylon with wool. A problem is introduced by this blend because of the difference in dye rate of the two fibers. Control of this problem is obtained through the introduction of specific retardants *(5)* and through the selection of colors. Color selection includes the use of neutral (metallized) and acid (metallized) colors, and the neutral dyeing and milling acid dyes.

A typical dyeing procedure for a hard twist yarn containing 30% nylon and 70% wool is as follows *(6)*:

The dyebath is set at 90° to 100°F with
0.5% Alkanol HCS surface-active agent
1.5% Nylon Resist GDC (a colorless, complex aromatic sulfonate)
5.0% ammonium acetate
and run for 10 minutes.

The pH is adjusted to 7.0 and the dyes are added.

Temperature is raised to 205°F in 60 minutes.

Dye one hour.

Acetic acid is used for exhaustion.

Colors which are recommended for dyeing nylon and wool blends by this procedure are listed in Table IV.

TABLE IV

Dyestuff	*CI No.*
Neutral Brown BGL	—
Acid Black 48	65005
Acid Red 80	68215
Acid Yellow 40	18950
Acid Green 25	61570
Acid Violet 43	60730
Acid Violet 34	61710
Acid Blue 58	—
Acid Blue 78	62105
Acid Blue 122	—
Acid Blue 113	26360

Combination carpet shades made from these dyes have been tested to give lightfastness of 40 to 80 Fade-Ometer hours.

Piece Dyeing. Piece dyeing of carpets has gained importance with the trend toward solid shades and the growth of the tufted approach to the manufacture of textile floor coverings. The advantages of piece dyeing are well known: special or custom shades are possible, more rapid order filling, lower yarn and carpet inventories, no dyed yarn or spinning waste. Nylon is extremely fortunate in that it readily lends itself to piece dyeing.

Tufted nylon carpets having widths up to 15 feet are being dyed commercially on specially designed dyebecks. Usually the 80-120 yard lots of carpet are dyed in open-width form. This is made possible by the use of front-roll spreading devices and side stop motions on the dyebecks. Most tufted nylon carpets are dyed before the latex backing is applied. Shading from side to center and end to end is a difficult problem encountered in the piece dyeing of wide carpets. An approach to overcoming this problem is to:

1) Maintain good circulation of the dyebath so that there are no large differences in dye concentration within the bath at any time.
2) Uniform temperature control of the dyebath. This may be partly obtained by using closed dyebecks heated at the top by use of a perforated steam sparger tube, and also by the uniform distribution of heating coils in the bath.
3) Running the carpet at a speed fast enough to insure rapid retreatment in the dyebath.
4) Proper dyeing technique involving the use of a nonionic surfactant, a long-chain alcohol sulfate, ammonia, and ammonium acetate in the dyebath when neutral (metallized) dyes are used.

A typical dyeing procedure for applying Capracyl neutral (metallized) dyes is as follows:

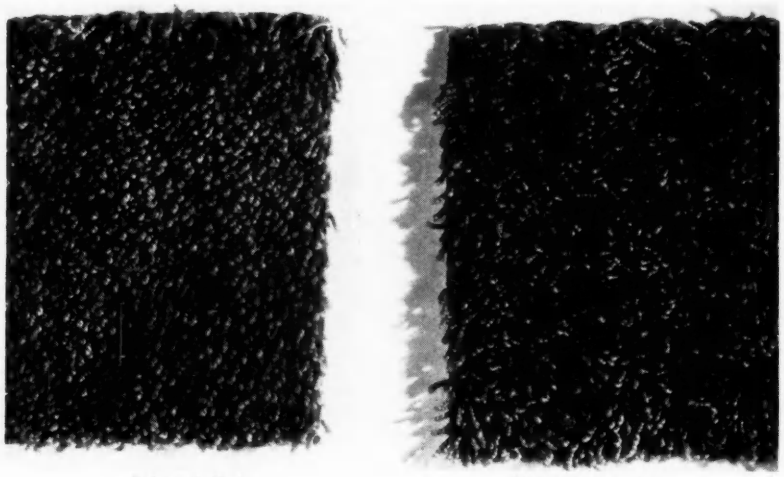
Set the bath at 80°F with:
2.0% Capracyl Leveling Salt
5.0% ammonium acetate
1.0% Duponol D Paste
1.0% ammonia (pH 7.5—8.0)

Run 10 minutes. Add the Capracyl dye and raise the temperature to the boil over a 45-minute period. Boil one hour and sample. It may be necessary to add additional ammonium acetate or acetic acid to obtain complete exhaust. A slow, cooling rinse is suggested. The use of an antistatic or softener finish is obviated, as they will generally accelerate the rate of carpet soiling during use. After dyeing, the carpet is open-width extracted and loop or frame dried before the latex backing is applied.

In cases where acceptable levelness cannot be obtained with neutral (metallized) dyes, it is suggested that the less fast disperse-type dyes be considered.

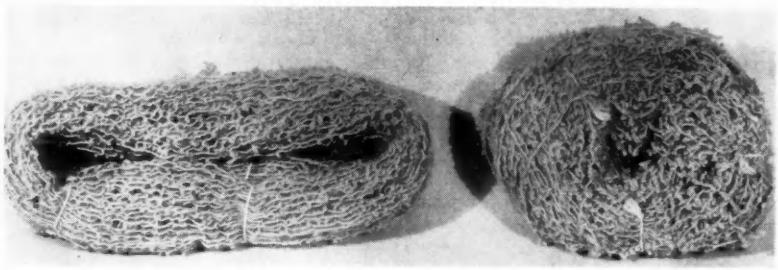
Additional methods for dyeing nylon carpets in piece form are under laboratory investigation or have been attempted in limited mill trials. They include the use of the Burlington Beam Dyeing Machine (7) and padbeck (6) dyeing techniques.

C) TWIST SETTING —— The ability of nylon to be shaped to a given configuration in the presence of heat is proving to be most useful by permitting carpets with durable textured effects. Carpets of nubby and frieze nylon yarns will retain their original appearance during long hard use and commercial cleaning because of nylon's matchless capacity to retain twist-set configurations.



Balanced Twist — Unbalanced Twist

Figure 3
Effect of twist



Before tumbling — After tumbling

Figure 4
Effect of skein tumbling

When nylon is subjected to conditions of heat setting or twist setting, it undergoes an internal molecular rearrangement or setting. The hydrogen bonding between the imido group of one chain and the carbonyl group of an adjacent chain, which is formed in the manufacture of nylon, is under considerable stress and strain. The setting operation causes these hydrogen bonds to sever and reform in a more relaxed position. These new bonds are much stronger than the original cross linkage and further rearrangement is most difficult. The presence of a swelling agent (moisture) during setting speeds the molecular rearrangement by penetrating the structure and assisting in the breaking of the cross bonds.

Types 100 and 600 nylon are used for yarns that are to be twist set. It should be remembered that the capacity of any nylon fiber for twist setting will be limited to some degree by previous exposures to live steam, hot water, or dry heat. Nylon carpet yarn is spun on all of the conventional yarn spinning systems. The staple count usually used in the cotton and woolen systems of spinning is 15 dpf, three-inch staple. Worsted spinning requires 15 dpf, 4½-inch or 6½-inch staple. With the American Worsted system, 15-denier Type 100 or 600 nylon tow is used. Flax spinning calls for 15-denier, 6½-inch or longer staple.

The techniques for twist setting and dyeing of nubby and frieze yarns made of 100% nylon are quite similar. The different appearance of these yarns is obtained through differences in yarn construction. For instance, a nubby yarn is usually prepared by plying two or more yarns to produce a balanced yarn which upon cutting stands erect. On the other hand, a frieze yarn gives a random appearance in a carpet because of the imbalance that exists in the component plied yarns (see Figure 3).

Comparative yarn constructions are listed in Table V.

TABLE V
Yarn construction of nubby versus frieze texture *(8)*

	Nubby	*Frieze*
Yarn size	2.5 to 4's cc/2 or 3 ply	2.5 to 4's cc/2 or 3 ply
Single twist	3.5 to 8.5 tpi, Z	2.5 to 4.0 tpi, Z
Ply twist	3 to 8 tpi, S	5 to 8 tpi, S

The desired effect may be varied by twist selection.

One of the important processing steps in making frieze yarn that is not required for nubby yarn is dry mechanical tumbling (about five minutes at room temperature). This develops the kink effect characteristic of frieze yarns. Figure 4 clearly illustrates this. Tumbling tends to balance out any differences in yarn caused by a variation in lag time on the twister bobbins.

Twist setting of nylon yarns may be carried out in either water or steam media. The choice will depend on the available equipment and the desired effect. In either case the desired texture must be evident in the yarn before setting because no alterations can be made once the yarn is set. The higher the temperature of twist setting, the better will be the twist retention and the deeper the dyeing. For this reason, variations in both texture retention and dyeability can be caused from batch to batch and within a batch by variations in setting temperatures. As a result of several programs aimed at determining the factors affecting the dyeability of twist set yarns, the following conclusions have been drawn:

1) Accurate temperature control during twist setting is essential to level dyeing.

2) Uniform temperature control is obtained more easily by the use of water than with steam. This is true because air pockets are eliminated and uniform heat transfer is obtained.

3) Competitive dyeing [neutral (metallized) dyes] of nylon frieze skeins twist set under a variety of conditions showed (Figure 5):

a) The higher the temperature used to twist set the yarn, the deeper the yarn will dye.

b) In the small batches tested, the time of twist setting ranging from 10 to 60 minutes had no apparent effect on dyeability at the same temperature.

c) Indications are that there is no effect of twist setting medium (water vs steam) on dyeability at the same temperature.

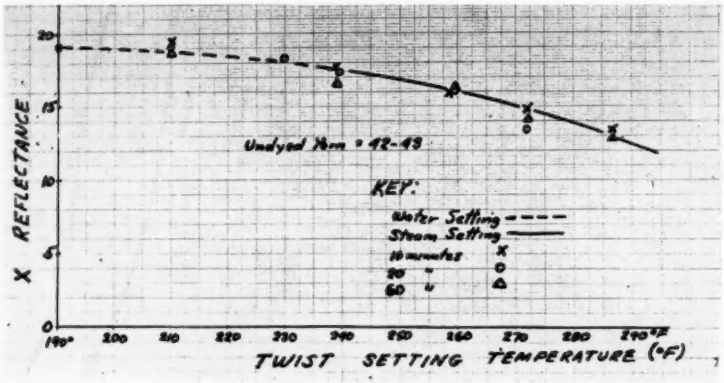


Figure 5
Effect of twist-setting conditions on dyeability of nylon frieze carpet yarn (red component)

D) WATER TWIST SETTING———Twist setting in water may be carried out at either pressure or atmospheric conditions. Pressure setting may be carried out in vessels, such as a pressure stock-dyeing kettle, and is preferred to atmospheric setting. Atmospheric setting, which is less complex since only boiling water is required, has been done in stock kettles and Hussong machines. Yarn which has been prepared from dyed stock will require pressure setting since, in this case, very little setting effect is obtained from atmospheric temperature. To prevent excessive color bleeding, a small amount of acetic acid (pH 6.0) is added to the setting bath. Commercial practice is to twist set for approximately one hour at temperature. Water twist setting causes a slight loss in twist definition because of the water flow through the yarn bundles. Another disadvantage of water setting is that an extracting and drying operation is required following the setting cycle.

E) STEAM TWIST SETTING———Twist setting with steam has the advantage of clearly defined yarn twist and no requirement of extracting or drying operations. In steam setting, it is very important that the pressure be uniform from load to load and that air be completely removed from the load prior to the high-temperature steaming operation. If air is not completely removed, the steam will not penetrate uniformly, resulting in possible variations in both texture and dye reception. The procedure in Table VI is typical of the vacuum-steam technique.

In the cycle outlined in Table VI, the purpose of the initial vacuum is to rid the vessel and yarn of all air to facilitate the penetration of steam. If air is present in the vessel, the penetration of heat into the innermost layers of yarn is impaired. Also, the presence of air during heating affects the yarn's dyeability by oxidizing and possibly yellowing the nylon.

The first steam should be introduced immediately after the initial vacuum to prevent air from re-entering the vessel. The four steaming periods assure uniform twist-setting temperatures throughout the load. The return to atmospheric pressure between steamings purges the spent steam prior to the introduction of new steam. Temperatures of 270°F throughout the load are obtainable with this technique. The final vacuum serves to cool the yarn and helps to remove the moisture of condensation.

When establishing the vacuum-steam cycle for any piece of equipment, it is suggested that thermocouples be used to determine and insure that the maximum temperature is achieved in the center of the load. Most external gauges do not record the temperature at the center of the yarn load.

Yarn made from dyed stock staple should be scoured prior to steam setting since some finishes have been known to cause a reduction in lightfastness of certain dyes when present on the yarn during the twist-setting operation.

One of the styling problems confronting the carpet manufacturer is to produce carpet with textured constructions having a good "see through" coverage with respect to the backing at a minimum cost. However, it is essential that yarn texture be retained even after extended periods of wear. This objective may be accomplished by several routes:

1) Use of minimum twist in the single yarns.

TABLE VI
Typical vacuum-steam cycle for twist setting

1) Vacuum (28" of mercury)
—Five to 12 minutes
2) Steam (28 psi gauge)
—Two minutes at pressure
3) Return almost to atmospheric (no vacuum)
—One-half to one minute normally required
4) Steam (28 psi gauge)
—Two minutes at pressure
5) Return almost to atmospheric (no vacuum)
—One-half to one minute normally required
6) Steam (28 psi gauge)
—Two minutes at pressure
7) Return almost to atmospheric (no vacuum)
—One-half to one minute normally required
8) Steam (28 psi gauge)
—Five to 10 minutes at pressure
9) Vacuum (28 inches of mercury)
—Six minutes

2) Partial setting of the twist so as to retain a large degree of twist, but permit a controlled amount of "bloom."

3) Use crimp set staple with unset staple to decrease the effectiveness of crimp setting on the yarn. This effect is demonstrated in Figure 6.

F) FINISHING———Tufted nylon carpets, after dyeing, extracting, drying, and latex backing, are inspected and shipped. Woven nylon carpets made from stock- or yarn-dyed nylon are given a light brushing and possibly a shearing. Many styles are merely inspected off the loom and shipped.

In the case of *loop pile* nylon carpet, the problem of pilling is sometimes encountered. The phenomenon of pilling can occur in fabrics of any staple fiber. However, if the fiber is strong and tough, as in the case of nylon, the pills do not get worn or broken off during normal use.

Recent laboratory work has defined the causes of pilling in nylon carpets and has resulted in the development of finishing procedures which will substantially reduce carpet fuzzing and pilling to an acceptable level (*9*). There are at least three factors that are responsible for the fuzzing and pilling of loop pile nylon carpets:

1) Inadequate binding of individual fibers in the tufts of pile yarn by the latex backing material.

2) Long or looped fibers in the pile yarn.

3) Initial surface fuzz.

Any one of these is sufficient to cause the carpet to pill or fuzz in use.

When the latex backing material does not thoroughly penetrate and tenaciously hold that part of the pile yarn which is in the backing, many fibers are free to work out of the yarn. Therefore, if each fiber is held securely in place by the latex, it cannot work to the carpet surface and pill.

When pile yarns contain fibers that

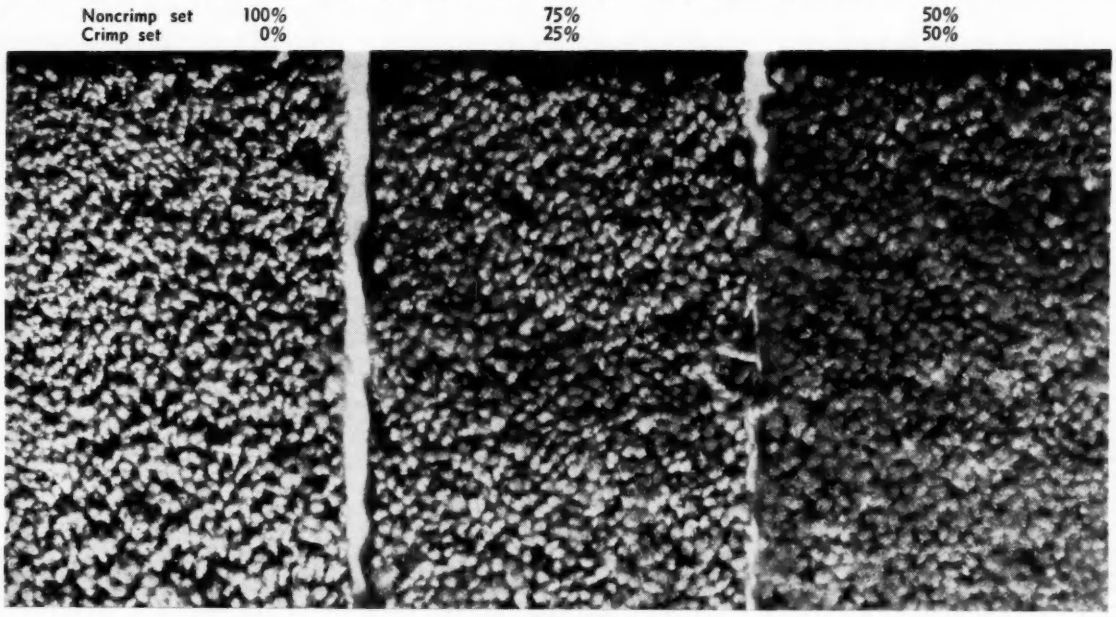


Figure 6
Effect of type of staple

are coiled, looped, or kinked in such a way that, if straightened, they are considerably longer than the tuft, pilling is likely to occur. Even though the kinked fibers are bound securely in the tufts, they are uncoiled and straightened by normal carpet wear. As they straighten, they work to the surface of the carpet making it fuzzy. Eventually the fibers become entangled, forming pills.

An investigation of several representative yarns showed that nylon yarns spun on the woolen system contained more long, looped fibers than nylon yarns spun on either the cotton or worsted system. One-inch sections were cut from yarn spun by these three systems. Then, the individual fibers were removed from the one-inch sections of the yarn, straightened, and measured. The percentage of fibers longer than one and one-half inch in these one-inch cuts is given in Table VII. Most of the action that produced these kinked or coiled fibers seems to occur during carding, and on the woolen card the rub apron aggravates the condition. It is likely that only the very longest of these "long" fibers participates in severe pilling.

A nylon carpet which has any degree of initial fuzz is likely to develop an objectionable or pilled surface during use.

G) CONTROLLING FUZZING AND PILLING——We have just discussed the factors contributing to the pilling of nylon carpets in wear. Further development work showed that it was possible to simulate the early stages of normal wear by a vigorous brushing of the fabric. This would pull out most of the loopy fibers that would be worked out during normal wear. If the resulting or raised fuzz is then removed, a carpet will result which has little or no tendency to pill in wear.

TABLE VII

Spinning system	*Fibers longer than 1½"*
Woolen	15%
Cotton	4%
Worsted	2%

All latices do not stick to nylon with the same degree of adhesion; therefore, it is important to chose the right one. Our tests have shown that GRS latices work best. No more than 200 parts of whiting or clay per 100 parts of latex solids should be used. Too much loading material reduces the binding power of the latex.

Once the fibers are locked in place by good application of latex, the next step is to raise the long, looped fibers from the pile yarn by brushing, and then to remove them by shearing. This is absolutely necessary with loop pile carpets made from *woolen* system yarns; it may not be necessary with some carpets made from yarns spun on the cotton or worsted systems, especially if the yarn twist is high.

An extensive evaluation of brushing conditions on both tufted and woven loop pile carpets of varying pile heights and densities indicates that two brushings are generally sufficient for all constructions. The conditions given in Table VIII have been found adequate for brushing densely constructed, low pile carpets.

TABLE VIII
Brushing conditions for loop pile carpets *(9)*

Brush clothing	34 gauge wire 70° angle straight tooth 1.125-inch staple Six rows (based on 1½" width clothing) Three twill 10 noggs
Brush speed	2400 ft/min at surface
Carpet speed	20 ft/min or less
Type of contact:	
Dense, low pile	"Point" contact with radius of curvature of dead rail one to two inches
Less dense, high pile	Point contact with radius of curvature of dead rail one-quarter inch or less
Nip force*	One to two and one-half lbs depending on carpet construction
Brushing sequence:	
Dense, low pile	Brush once in each direction
Less dense, high pile	Brush twice in same direction

*Nip force is determined by measuring the tangential force required to draw an eight and one-half inch wide strip of Bristol paper, 0.007 inch thick, through the nip of the brush against the carpet while the brush is not rotating. The paper is drawn through the nip in the direction which is against the rotation of the brush.

The long, looped fibers that have been raised by brushing are sheared off by passing the carpet several times through a well-adjusted, well-sharpened shear.

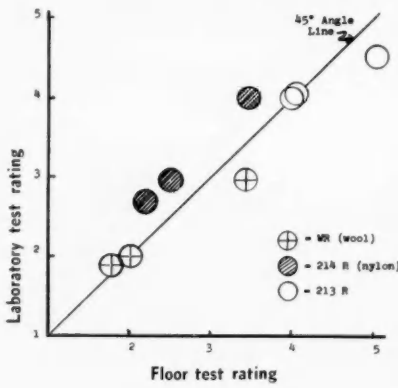


Figure 7
Correlation of laboratory and floor tests for pilling

H) TESTING FOR FUZZING AND PILLING———One way to determine the fuzzing or pilling tendency of a carpet is to place the carpet on the floor where it will get considerable traffic and to observe its performance over a period of time. Locating test samples under desks where there is extensive shuffling of feet is especially likely to accelerate fuzzing and pilling.

Because floor testing requires such a great amount of time, an accelerated laboratory method has been developed that can be used to predict fuzzing and pilling tendencies of carpets in a comparatively short time. The method consists of tumbling carpet squares in an unheated home-type clothes dryer with a number of rubber strips and balls of fabric, which act as abradants. The fabric balls also supply a small amount of fine lint. During tumbling, pills are produced on the carpet squares that resemble those produced in actual service.

Good correlation has been obtained between laboratory tests and actual floor service both in the degree of pilling and the difference of the pills and fuzz. A series of carpets was floor tested in a hallway for a total of 16,000 traffics and also tested in the laboratory machine for five hours. Several persons rated both sets of samples. The ratings were plotted in Figure 7. In under-desk service, the ratings obtained on these carpets were slightly higher; however, they were in the same general order.

The procedure for laboratory pill testing carpets is to cut the specimens into eight-inch squares.[1] Two such carpet squares are then stapled together back to back[2] with a piece of cardboard between them to act as a stiffener and prevent curling.

Six to eight of these "sandwiches" are loaded into the drum of a tumble dryer that is equipped to blow unheated air through the drum.[3] The dryer is also loaded with the following items:

30 strips of natural rubber sheet (Durometer A[4] hardness about 40) (18½″ x 5¾″ x 3/32″) — eight lbs.

Five strips of Neoprene sheet (Durometer A hardness about 50) (17¾″ x 5¾″ x ⅛″). Each Neoprene strip has 20 to 30 one-inch diameter holes cut into it to increase the snagging type of abrasion—three lbs.

Six balls of tightly rolled cotton rags (five inches in diameter)—three lbs.

This load, which weighs about 20 pounds, is then tumbled for 10 hours. In the laboratory tests, 10 hours in the tumble tester has been accepted

[1] The size apparently is not critical; specimens ranging from six to 10″ square have been tested with no apparent differences.

[2] Bostitch Model C stapler using staples having a one-half-inch leg.

[3] We have used a Norge Model AE-620. This machine has a nonperforated drum in which air enters at the back and leaves at the front. The drum diameter is 29″ and the speed or rotation is 40 rpm. The regular door was replaced with one made of clear plastic to permit observation of the tumbling. The time switch was replaced with a toggle on-off switch. Undoubtedly, other clothes dryers could be adapted to do the same job.

[4] Short Instrument & Mfg Co, Jamaica, NY.

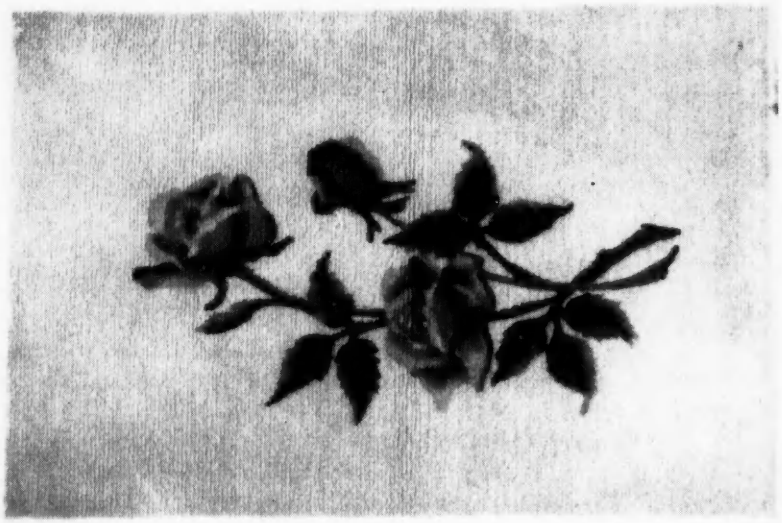


Figure 8

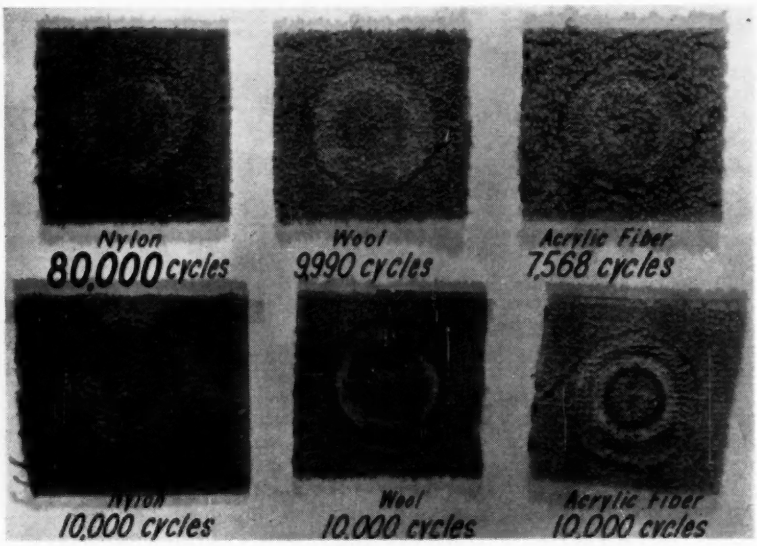


Figure 9
Abrasion test samples

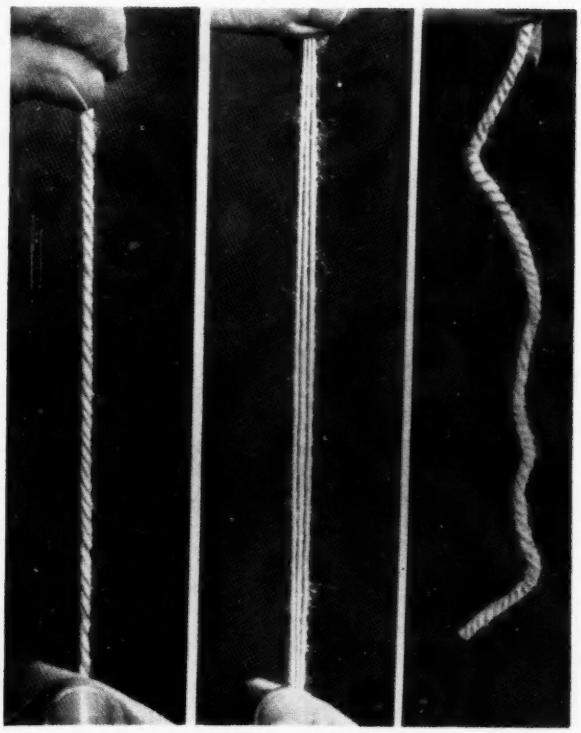


Figure 10

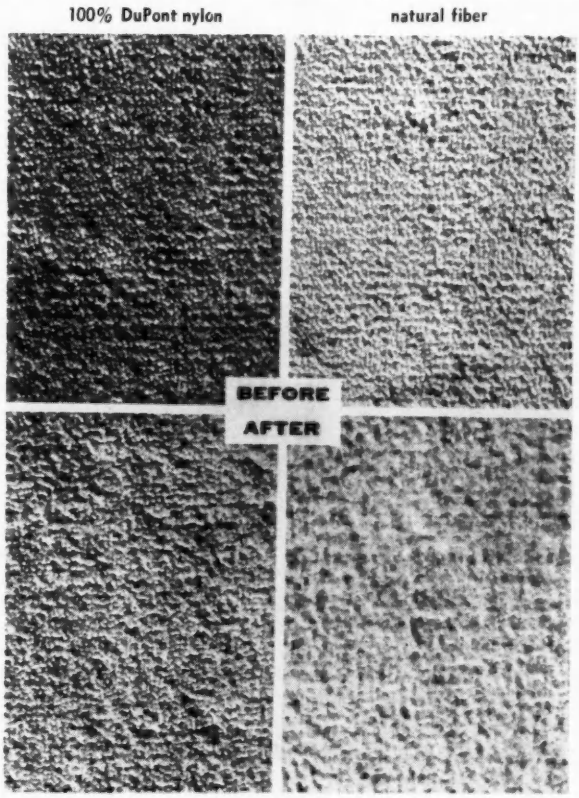


Figure 11
Effect of scrubbing with an oscillating scrubbing machine

as approximately the equivalent of a 32,000 traffic level in floor testing. At the end of the tumbling period, the samples are removed, unstapled, the edges trimmed and beveled, and the surface brushed lightly to remove loose lint and to erect the pills. The samples are then evaluated subjectively by a panel of several persons. First, each person ranks the specimens within a test lot according to the amount of pilling and then rates each specimen on an absolute scale for pilling and fuzzing by the following descriptive system:

1—none
2—slight
3—moderate
4—bad
5—extremely bad

Fractional ratings can be given. On this scale, a rating of 2.0 to 2.5 represents the borderline of acceptability.

I) PRINTING———The printing of 15-foot-width nylon carpet is a development being perfected by "Deep Dye Industries", Inc, New York, which will permit greater styling flexibility of nylon carpet, especially tufted carpet. U S and foreign patents are pending. We understand that the technique and equipment will be licensed on a royalty basis to carpet manufacturers or commission dyers. Examples showing the ability of nylon carpet to be printed with clearly defined patterns and excellent penetration in multiple colors are shown in Figure 8.

J) TESTING OF CARPET PERFORMANCE———The performance of nylon in carpets has been demonstrated in several ways over the years. However, it may be of interest to discuss a few of the performance tests which have been used in characterizing nylon for carpets. These may possibly be used for evaluating other carpet fibers. A few of the important attributes of any fiber in carpets are:

1) Wear life.
2) Recovery from crushing by furniture.
3) Texture retention.
4) Recovery from crushing by foot steps.
5) Cleanability and spot removal.
6) Dyefastness.

These will be discussed separately in the following paragraphs.

1) *Wear Life.* In the laboratory there are three tests which help guide the thinking as to the possible wear life of a carpet fiber. These are: Taber abrader, flex tester, and floor tests. The abrader and floor tests are generally conducted by using samples of carpet, while the flex abrader is used to determine the flex life of yarns.

In actual flat abrasion tests made on the Taber abrader, nylon lasted more than five times as long as the next best carpet fiber. This is shown in Figure 9.

The flex life of nylon yarn was demonstrated to withstand 750,000 flex cycles. Tests have shown this is more than 15 times more cycles than the next best natural fiber and 24 times more than the best of other man-made fibers now used in carpets.

Laboratory tests on carpet and yarn can only indicate the performance of a fiber in actual use. For this reason many nylon carpets are actually floor tested. As an example, a 100% nylon carpet which was installed in a cafeteria outwore the traditional carpet by 19 times. In another recorded wear test, a nylon carpet was placed in use on a concrete floor at the entrance to an industrial plant. Today, after two million traffics (approximately 20 years' wear in the

average home), the carpet is still in service.

2) *Recovery from Crushing by Furniture.* The "dead weight" test is used in the laboratory to measure the propensity of carpets to recover from crushing by furniture. This consists of weights so arranged as to impart 19 pounds per square inch to test samples of carpet. The test is carried out for different times, but generally 18 hours is sufficient. Following the "dead weight" test, the thickness of the fabric is measured with a compressometer after conditioning 48 hours. It was found that nylon carpets had the best recovery of any fiber commonly used in carpet at an average recovery of 95.9%.

3) *Texture Retention.* When nylon carpet yarns are subjected to heat and moisture, they become set and develop a "memory" for returning to the set condition. Figure 10 shows that a three-ply nylon yarn, when untwisted, will quickly spring back to its original highly twisted state when relaxed. This property of nylon contributes to texture retention never before known in a carpet fiber.

Wet cleaning is a critical test for determining a carpet's ability to retain its textured appearance. Figure 11 shows samples of equivalent natural fiber and nylon constructions after five wet scrubbings with an automatic oscillating scrubbing machine. Nylon has retained most of its original texture while the control natural fiber has become matted and lifeless.

4) *Recovery from Crushing by Foot Steps.* In the laboratory, the Walker Crush Tester simulates actual crush conditions of heavy foot traffic on an accelerated basis. Pile height of the carpet is measured before the test and at intervals throughout the entire 2,000 compression cycles. The 2,000 cycles will correlate with 30,000 - 40,000 foot traffics. Measurements of height are made on a compressometer. In this test, the pile height recovery for 100% nylon averaged 94.5% for loop pile, 91.2% for cut pile; while the next best test item containing a natural fiber averaged 91.2% for the loop pile and 88.0% for cut pile.

In a test (Figure 12) using 75,000 actual foot traffics, nylon retained 83%, wool 67%, and the acrylic 62% of their original pile height.

5) *Cleaning and Spot Removal.* The cleaning and spot removal performance of nylon is one of its outstanding attributes. It is difficult for stains to penetrate the fiber, and because of the smooth surface, stains do not become very firmly attached. To dramatically prove the ruggedness and easy cleanability of nylon carpets, a section of 100% nylon carpet was riveted to a road leading to the Du Pont Experimental Station. In 43 days of the test, 25,191 passenger cars, delivery vans, trucks, and construction equipment passed over the carpet. The conditions were the worst possible with freezing rain and snow and the temperature fell as low as 17°F. Sections of the carpet which had become covered with grease and stains of all kinds, were beautifully cleaned, retaining the texture and approaching their original appearance. Many equally dramatic stories have been developed by stores all over the country.

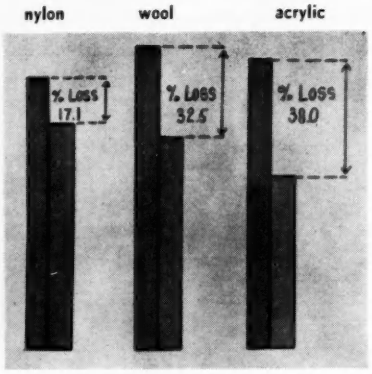


Figure 12
Recovery from crushing by footsteps before and after 75,000 traffics

Nylon's excellent cleanability with regard to the typical household stains such as catsup, nail polish, lipstick, mustard, coffee, cola, etc, has been clearly demonstrated in actual use as well as in the laboratory (*10*).

6) *Colorfastness.* Nylon has colorfastness of 40 to 80 Fade-Ometer hours when the best acid dyes are used. The wetfastness of nylon in carpet has proven to be adequate. Typical mill tests for bleeding, spotting and washing have shown that nylon will meet all consumer cleaning requirements.

One should conclude that there are numerous tests for determining the performance characteristics of carpet fibers and that nylon's performance as a carpet fiber is excellent.

K) CONSUMER ACCEPTANCE OF 100% NYLON CARPETING ———During 1957, a marketing research organization (*10*) conducted a survey among the owners of carpets made of 100% nylon. The objective of the survey was to determine from the carpet owners just what they thought of the nylon carpets and if their next carpet purchased would be nylon. Approximately 146 owners who had used their carpets for approximately 18 months responded.

Questions such as the following were asked:

1) How well has the appearance of your carpet held up?

144 replied:

Extremely well pleased	56%
Satisfactorily	36%
Not satisfactorily	8%

2) Please list what you believe to be the important advantages and disadvantages of your 100% nylon carpet.

139 consumers gave 333 statements of advantages and 84 statements of disadvantages:

Advantages (333 statements)		*Disadvantages (84 statements)*	
Cleans easily	45%	Matting	13%
Wears well	38%	Static electricity	11%
Spotcleans easily	27%	Difficult to clean	8%
Moth repellent	24%	Stretches	5%
Doesn't mat	22%	Lint adheres	4%
Attractive appearance	17%	Others (none over 3%)	26%
Others	12%		

3) Have you ever spot cleaned your carpet?

146 answered as follows:

Yes	—	81%
No	—	19%

4) If yes, how successful was the cleaning?

118 answered as follows:

Very disappointed	0%
Disappointed	4%
Satisfied	44%
Extremely well pleased	52%

5) Please give your over-all evaluation of your 100% nylon carpet.

145 answered as follows:

Too soon to judge	1%
Extremely disappointed	1%
Dissatisfied	4%
Satisfied	32%
Very well pleased	62%

6) Based on your current knowledge of carpet fiber, what kind of carpet do you expect to purchase in the future?

126 answered as follows:

100% nylon	76%
100% wool	13%
Wool/nylon blend	8%
Others	3%
Cotton	0%
Rayon	0%

We conclude that the results of this survey indicate that nylon carpets are headed for increasing consumer acceptance.

TRADEMARKS

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LITERATURE CITED

(1) Technical Information (du Pont) Textile Fibers Bulletin N-29 (October 1955).
(2) *Technical Bull (du Pont)* **11**, No. 1, 37 (March, 1955).
(3) Fletcher, J M, *Am Dyestuff Reptr* **46**, No. 9, 319 (May 6, 1957).
(4) *Tech Bull (du Pont)* **11**, No. 3, 122 (September, 1955).
(5) Muller, C E, and Nusslein Jr, U S Pat 2,325,972, General Aniline and Film Corp (1943).
(6) Wygand, W J, *Am Dyestuff Reptr* **46**, No. 26, P988 (December 30, 1957).
(7) Meunier, P L, *ibid* **45**, No. 11, P327 (May 21, 1956).
(8) Technical Information (du Pont) Textile Fibers Bulletin N-86 (February 1957).
(9) Technical Information (du Pont) Textile Fibers Bulletin N-87 (March 1957).
(10) Burnam, C W, *National Rug Cleaner* (November 1955).

QUESTIONS & ANSWERS

Question: The carpet trade, over a number of years, has established that, for wool, it is desirable to have not only a certain average fiber width, but that for certain aspects of cover, that this size shall have a certain distribution.

Do you recommend the use of 15-denier nylon only, or are you recommending that a variety of deniers be blended?

Answer: Our tests indicated that 15-denier is best. I don't want to get into a discussion about what is good for wool, and what is good for nylon. But, certainly, in wool, you take the natural product and use it to your best advantage. In nylon, we take the same position. We provide the best product we can for the carpet.

We feel that, of presently available products, 15-denier is the best. This is not to say that there will never be improvements; however, right now we are pretty close to an ideal carpet denier with our 15-denier fiber.

Q: Are you making any specific recommendations to the carpet manufacturers, with reference to application of finishes?

A: There is always a problem of putting finishes on the carpet and increasing the soiling. You get out of one trouble, and into a worse trouble sometimes.

Q: In the comparison of nylon versus wool, the pile was about the same height for the nylon as for the wool. How did the performance compare and what would be the price differential?

A: We were not comparing nylon with wool. We had one question that asked: "Based on your current knowledge of carpet fiber, what kind of carpet would you buy next time?" These people who owned nylon carpet were also asked: "How well do you like them?"

I presume that perhaps they did have a wool carpet in their homes; however, the comparison question was not asked.

Q: What is your preferred way of piece-dyeing the carpets? I understand that some of them are done with continuous dyeing methods.

A: I did some work in dyeing on beam equipment, but I believe that method is not being used very extensively right now, probably because of the pile crushing and possibly other reasons.

I believe if you compare the dyebox with beam dyeing equipment, the dyebox is being used much more extensively.

Now, let us switch over to continuous dyeing. That lends itself to the large lot type of operation. It is something that is growing. It is good, and it is here to stay. The people who are dyeing automotive fabrics are doing a lot of continuous dyeing, where they can use the narrow width, and padders are being made that would handle wide carpeting. But I believe that, right now, not much is being done on the continuous dyeing of the wide carpeting, except in the rayon field.

Q: Would you explain about crimp setting the fiber, and also twist setting? The crimp setting is done on the individual staple, and the twist setting is done by the yarn spinners on yarn?

A: That is right. We do the crimp setting at our plant. The people in stock dyeing do the crimp setting, too, because they can buy it uncrimped, and it will get crimped in the dyeing operation.

After yarn is made into an entity, you set the twist to shape it.

Q: Are you in a position to give some rough figures on the distribution of worsted spun, cotton spun and woolen spun nylon yarns in carpets?

A: No, I cannot give you a percentage. I could say that there is a considerable amount spun on the cotton system, and also a considerable amount spun on the worsted system.

On the woolen system, I would say that that is the lowest volume of the three.

It has been pointed out that the worsted and the cotton systems do have less tendency to fuzz and pill.

We do not want to give the impression that the woolen system is not satisfactory, because we have a current bulletin on the woolen system, and I could give you a copy of that.

Q: I recall vaguely that the greater recovery of nylon fiber is much slower than some of the other fibers, although the recovery is good, and it takes longer to do that.

I also recollect that nylon carpeting, in general, has a lower pile than woolen carpets. Is that because they tend to muss or deform more readily than the woolen carpet?

A: We hear a multitude of reports on the recovery of nylon as compared to wool, but when we actually run the tests, we are surprised at the good permanence.

One test is the immediate recovery test, which simulates the effect of footsteps. Another is recovery from a dead weight load like that imparted by a piano setting on a carpet for several months. In both of these tests, nylon has performed exceedingly well compared with wool.

There is another reason for a lower pile and that is excellent durability; you don't need as much fiber. The cost is also considered in the carpet construction.

Q: What is the method of twist setting? You showed temperatures up to 290 degrees.

A: There are two methods that are used commercially. One is in water under pressure, and it doesn't seem to make too much difference whether you use steam or water. You should get around 250°F or something like that to get good twist retention in the carpet yarns.

The people who do not have a dryer prefer to use steam, and there they use the automatic vacuum steam type of apparatus. You must get the air out in order to get the steam in. That is one of the serious problems. Be sure to get all of the air out, in order that the yarn can be uniformly set.

Exposure of the yarn to around 250°F or 260°F for a fairly short time is sufficient to obtain setting.

Technical Committee on Research———

THE STATISTICAL APPROACH IN DEVELOPING TEST METHODS*

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Statistician, Engineering Dept
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INTRODUCTION

MOST test methods are developed to fill a certain need for maintaining some quality level which often determines the suitability of an article for the end-use intended. The actual suitability under end-use conditions sometimes cannot be measured and, if measurable, the time element and economics involved does not usually permit it. Physical tests must be developed to serve as indicators of how the items will perform under end-use conditions. Again, because of the time element, the test in the laboratory is an attempt to accelerate the conditions encountered in service. The trick is to accomplish the acceleration without causing a reversal in the scale which associates end-use performance with the physical measurement. Very often the method of accelerating the physical measurement may cause something different to be measured in the laboratory which will not accurately predict service performance in every instance. A statistical correlation does not necessarily mean that the relationship is casual.

To be specific, let us examine a test which most of you are familiar with—Damage Caused by Retained Chlorine.

The objective is to develop an accelerated laboratory test to predict the behavior of fabrics with respect to tensile loss when exposed to chlorine washes under service conditions.

A test procedure was recommended which simulated exposure to chlorine under service conditions except that acceleration of conditions was attempted by increasing the chlorine concentration 10-20 fold over the amounts generally employed in commercial and home washes. This procedure correlated with the history of some fabrics in actual service. Interlaboratory tests were then initiated to check the reproducibility of the test and the results were discouraging. There was little or no agreement between laboratories. There was indication, however, that repeated tests conducted in the same laboratory gave better reproducibility than tests conducted in different laboratories.

TABLE I
Percent loss in tensile due to retained chlorine
Laboratories

	A	*B*	*C*	*D*
Fabric I	18	63	35	0
	23	54	42	10
	16	71	32	−4
	19	59	47	3
	25	68	45	8
Fabric II	47	22	27	65
	32	20	25	52
	39	15	19	57
	45	17	29	66
	30	25	20	48

Table I shows results which are typical of some of the data obtained in interlaboratory tests.

The results are given for four laboratories with each laboratory making five separate determinations of percent loss in tensile strength due to retained chlorine on each of two fabrics. From Table I we see that laboratory A obtained losses of 20% for Fabric I while laboratory B obtained losses of 60% for the same fabric. Statistically, this could only mean that certain important factors which influence the numbers obtained in the measurements had not been taken into account. The discrepancies cannot be attributed to bias since the laboratories obtained results for Fabric II which show reversals in the ranking. For example, laboratory A obtained higher losses for Fabric II than did laboratory B. The differences could not be ascribed to experimental errors of equipment or operation, since the repeat measurements made close together in any one laboratory indicated good agreement.

To avoid such apparent discrepancies in what may still be a valid laboratory test procedure, it is recommended that, in the early stages of developing a test method, effort be concentrated in one laboratory having a qualified operator working with proper equipment in steady surroundings.

At this early stage all the factors which may influence the laboratory measurement should be enumerated and statistically investigated.

For example, with the chlorine retention problem we might start to list the factors as follows:

X_1 Concentration of available chlorine
X_2 Liquor-to-cloth ratio
X_3 Temperature of chlorination bath
X_4 pH of chlorination bath
X_5 Time of exposure in bath
X_6 Temperature of scorch tester

Mathematically, we can state that the percent loss in tensile strength, y_1, for a particular class of fabric is some function of the variables listed:

$$y_1 = f(x_1, x_2, x_3, x_4, \ldots x_n).$$

There would be a similar relationship for all other classes of fabric which are essentially different from one another:

$$y_2 = f(x_1, x_2, x_3, x_4, \ldots x_n)$$
$$y_3 = f(x_1, x_2, x_3, x_4, \ldots x_n)$$
$$\vdots$$
$$y_k = f(x_1, x_2, x_3, x_4, \ldots x_n).$$

To thoroughly investigate all the variables to approximately define these relationships without a statistical approach would require a prohibitive number of experiments and the project would be defeated at the start. Using a statistical design, as many as 10 variables can be screened for importance with as little as 32 experiments (*1*): The important factors can then be explored in more detail with subsequently designed experiments which are more thorough. The result would permit an understanding of the operation of the system with the quantitative effects of the important variables of the original relationship known approximately.

A byproduct of the analysis would be a measure of the reproducibility of the method. The significance of each factor would also be obtained

*Presented before the 195th meeting of the Technical Committee on Research on May 22, 1958 at the Sheraton Hotel, Philadelphia, Pa.

with an objective regard for the errors due to experimentation.

When the relationship between the variables and the laboratory determination of chlorine damage is known with reasonable assurance, the next step in the test development is to specify the level of each factor that will constitute the test procedure.

The levels must be chosen so that a) the test procedure that is specified will correlate with information obtained from service testing, and b) the test will be an accelerated test that will provide the necessary prediction of service performance in a reasonable time.

CORRELATION WITH SERVICE TESTING

The problem of correlation is truly a statistical problem. A first step is to collect data from fabrics tested under use conditions. The fabrics selected for use study should include all items from the more important categories of fabric on the market which are essentially different from one another either in preparation or finish which were used in the laboratory study. Some categories suggested are:

Untreated cotton, dyed
Untreated cotton, undyed
Treated cotton, urea-formaldehyde resin
Treated cotton, cyclic urea-formaldehyde resin
Treated cotton, melamine-formaldehyde resin
Untreated rayon, dyed, etc.

The Committee on Damage Caused by Retained Chlorine would find it useful to set up some criterion for sample selection which would keep the number of categories down to a reasonable number. Since specimens of these same fabrics were used in the statistical study of the laboratory test procedure, the tensile loss—variable relationship would also be known for each fabric type.

Knowing the results of the service tests and knowing the laboratory relationship between tensile loss and test variables, the levels of all the laboratory variables may then be set at specified levels which would give the best correlation between laboratory tensile loss due to chlorine retention and the tensile losses obtained in use testing.

To illustrate, suppose the following relationships were obtained in the laboratory study:

	Use test % loss
$y_1 = 18 + 6x_1 - 7x_2 + 18x_3 - 2x_4 \ldots + 3x_n$	80
$y_2 = 12 - 2x_1 + 4x_2 + 21x_3 + 3x_4 \ldots + 1.5x_n$	65
$y_3 = 3 + 11x_1 - 6x_2 + 9x_3 + 4x_4 \ldots - 2x_n$	0
. . .	. . .
$y_k = 7 + 3x_1 + 5x_2 + 25x_3 - 7x_4 \ldots + 7x_n$	17

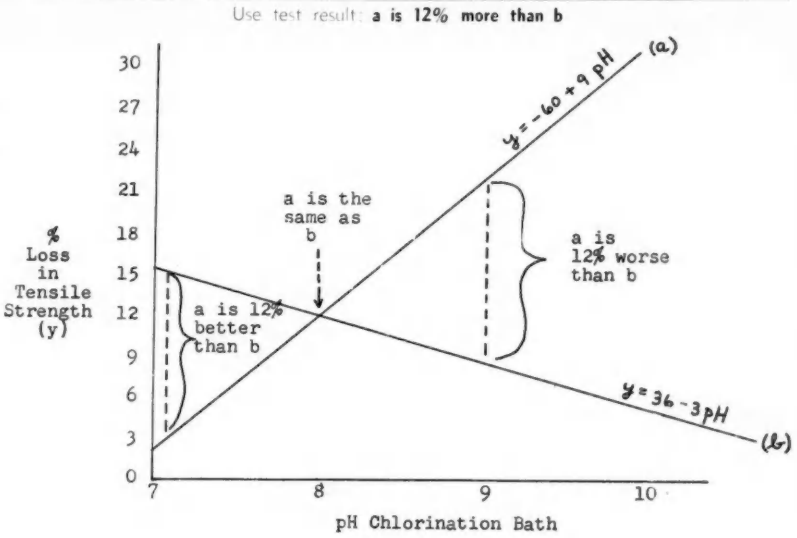

Figure 1

If the number of fabric items, k, is greater than the number of laboratory variables, n, we can substitute the service testing data for y_1, y_2, . . . yk in the expressions and solve the resulting simultaneous linear equations for the values of x_1, x_2, x_3 . . . x_n which would correlate the laboratory data with the use test data. A requirement is that the levels of the variables should not be selected outside of the experimental area investigated. Other restrictions may have to be included which would tend to make the problem more complex. However, linear programming (2) techniques may be employed to obtain the solution.

Let us consider a simple example to further explain the method of obtaining the correlation with use test data. Suppose there were only two fabric items (a, b) which were of interest and only one important variable in the laboratory test procedure, the pH of the chlorination bath.

As a result of the laboratory statistical study let us assume the following relationships were obtained:

$$y\ (a) = -60 + 9\ pH$$
$$y\ (b) = 36 - 3\ pH$$

Figure 1 shows these two relationships plotted. Let us suppose that the use test results indicated fabric "a" to experience a 12% higher loss in tensile strength than fabric "b". From Figure 1 we can see that, at a pH of slightly greater than 7, the opposite is true, ie, fabric "a" is 12% better than fabric "b". At a pH of 8, both fabrics display the same loss in tensile strength; whereas at a pH of 9, fabric "a" is 12% worse than fabric "b". Obviously, the level of pH to recommend for the laboratory test procedure is 9, for at this pH a correlation with end-use data is obtained.

There is one word of caution; the association between the observed service values and the laboratory test values would be strictly a statistical relationship, and nothing more. Though the correlation may be used as an estimate of a functional or causal relationship, misleading results may sometimes be obtained. For example, a category of fabric not previously tested may display an entirely different relationship to the laboratory test variables and thus the predicted loss by the laboratory test may not agree with serviceability history.

In any event, once the test method is completely specified, an interlaboratory test is in order to check the reproducibility of the test method. If all the important variables have been studied previously and set at recommended levels, then the laboratories have a good chance to agree within limits of experimental error *if instructions have been followed closely.* Here again, a statistically designed experiment can serve to answer the following questions:

1) How reproducible is the proposed test method in view of the laboratory to laboratory differences obtained in the interlaboratory study?
2) How many specimens should be run in order to be reasonably sure that the test result is known within certain specified tolerance limits?
3) Is there an additional important variable in the test procedure which has not been considered

in the initial study? This would be evident if the laboratory to laboratory variation was significantly higher than the variation within laboratories.

4) Other questions which may be of interest which can be included in the design of experiments.

An example of employing a statistical design in an interlaboratory test to answer specific questions was the Accelerotor study run about a year ago as part of the program by the Committee on Abrasion. The results answered the questions raised concerning:

1) Reproducibility of the Accelerotor.
2) Significance of abrasive liner wear on successive samples run in the Accelerotor.
3) Correlation with service data on several fabrics whose service history was known.
4) Machine variability so that the number of swatches to run could be specified for obtaining results with any desired precision.

From the uses cited we see that statistics deals with two difficult areas in developing test methods—variation and complex relationships. In developing test methods, inferences and decisions are based on sets of data. It is characteristic of such data that a similar set obtained under substantially identical conditions is different, —this is variation.

Likewise, it is a rare event that a physical measurement such as chlorine damage or resistance to abrasion is dependent upon a single factor. Usually these quantities are the result of the complex interaction of several factors. It is the purpose of statistics to design economical methods for observing systems and analyzing the results so that it is possible, despite variation, to obtain an insight into the complex relationship. Having a better understanding of the operation of the system, the way to achieving the original objectives of the test method is made clear.

It was mentioned earlier that, under end-use conditions, the performance of material may be objectively immeasurable in certain cases. One such area would be in evaluating a fabric for abrasion resistance by service testing. To develop good abrasion testing machines in the laboratory, it is necessary to have some serviceability data to use as a basis for correlation.

A statistical method which might find utility in this and similar areas is the technique developed by Kendall (*3*) employing paired comparisons. Suppose you were interested in obtaining wear data on five items to rank the items according to their resistance to abrasion. It is difficult to evaluate the items if they are handed out one to a subject even though replicate samples are employed. This stems mainly from the fact that there is a tremendous difference in the amount of abuse a garment will receive, depending on the individual wearer. This variation is usually so great that it obscures any differences in abrasion resistance which may exist between items. An alternate method is one in which garments are rotated so that each subject has the opportunity to wear each of the items an equal length of time. Selection of subjects according to size and the difficulty in obtaining judgments about the final ranking of items is encountered.

In the method of paired comparisons, two items (or more) are distributed to each subject. For five items there are $\frac{5(5-1)}{2}$ possible pairs to evaluate if each item is to be compared with every other item once, ie:

1–2	2–4
1–3	2–5
1–4	3–4
1–5	3–5
2–3	4–5

Each subject would alternately wear each of the two garments until a difference in wearing quality is noted or for a specified duration of time, whereupon a preference is made for one of the items in the pair. The preferences from all the judges are then tabulated in the following form:

	Items 1	2	3	4	5	*Row Sum*
1	–	1	1	0	0	2
2	0	–	0	0	1	1
3	0	1	–	0	0	1
4	1	1	1	–	0	3
5	1	0	1	1	–	3

The entry of 1 in the first row, second column means that, on the occasion when the pair 1-2 was evaluated, the first item displayed better abrasion resistance than the second item. An entry of zero would have indicated that item one was not preferred. The row sum indicates how the items are ranked.

In the above tabulation there are some discrepancies. For example, we see that 4 is preferred to 2, and 2 is preferred to 5, but 5 is preferred to 4! Kendall's method weights these discrepancies to obtain a final ranking of the items being compared.

Of course, in practice, we may want to run more than one replicate for each possible paired comparison to obtain significant differences between rankings. Along this line, a method for calculating maximum likelihood probabilities for each item in this paired comparison scheme have been developed by Bradley and Terry (*4*). As a result, given a pair of any two items we can state the probability of one item being preferred over the other by a subject randomly selected from the population. The probability that the obtained ranking of items could be obtained by chance when there are really no differences between items can also be computed. Tables have been published (*4*) giving these probabilities up to five items and five replications.

A more elaborate scheme which includes a seven-point scale to denote preference has been devised by Scheffe (*5*). The method permits an analysis of variance to be performed on the data to detect significant differences between items.

The advantages in running service tests by paired comparisons are:

1) It permits evaluation of items on a scale even though the preference is purely subjective.
2) The method is not affected by variability between wearers.
3) Items are ranked with comparative ease even though there is variation between duplicate items.

SUMMARY

A few concepts of statistical design that are appropriate for use in the development of new test methods have been given. A point to be emphasized is that any statistical design is tailor-made to answer the specific questions raised by a consideration of the over-all objective. Therefore, the time to call in a statistician is not after data have been collected but rather at the start. Once the proper plan for collecting data is resolved, the analysis is usually straightforward.

REFERENCES

(*1*) "Fractional Factorial Experiment Designs for Factors at Two Levels," U S Department of Commerce, National Bureau of Standards, Applied Mathematics Series—48.
(*2*) Vajda, S, "Linear Programming," Wiley, 1956.
(*3*) Kendall, M G, "Further Contributions to the Theory of Paired Comparisons," *Biometrics* (March, 1955).
(*4*) Bradley, R A, and Terry, M E, "The Rank Analysis of Incomplete Block Designs, I," Biannual Report No. 2, Statistical Methods for Sensory Difference Tests of Food Quality, published by the Statistical Laboratory, Virginia Polytechnic Institute, Blacksburg, Va.
(*5*) Scheffe, H, "An Analysis of Variance for Paired Comparisons," *J Am Stat Assoc* (Sept, 1952).

Minutes———

TECHNICAL COMMITTEE ON RESEARCH
195th Meeting

THE 195th meeting of the Technical Committee on Research was convened at 2 pm in the Independence Room of the Sheraton Hotel, Philadelphia Pa, on Thursday, May 22, 1958, with the following members present:

C A Sylvester, chairman of TCR
H W Stiegler, director of research
C W Dorn, chairman of ECR

W D Appel	L S Little
C A Baker	H E Millson
V L Chase	J E Norton
A T Clifford	B R Norwick
B K Easton	W F Prien
S M Edelstein	F A Prisley
W E Fayssoux	F J Rizzo
A H Gaede	J W Schappel
H B Goldstein	W M Scott
A E Hager	C A Seibert
V B Holland	R L Stutz
A I Hultberg	G M Smith
W A Holst	L S Thompson
R W Jacoby	E G Wiest
A E Johnson	H E Wilde
E R Kaswell	M W Winkler

C L Zimmerman

Others present included:

R E Derby	T D Miles
H E Glidden	E Morrill
G M Kidder	G M Richardson
W Klaas	V S Salvin
J F Kosowicz	H G Smith
G J Mandikos	F J Szurek

F V Traut

After delivering his opening remarks, TCR Chairman Sylvester called upon Charles W Dorn, chairman of the Executive Committee on Research, for a brief report. Mr Dorn stated that there would not be much to report because, under the new arrangements, ECR does not meet until after the TCR meeting. This permits ECR to take immediate action on any committee requests or business requirements that stem from the TCR meeting. He stated that several important policy items were expected to be on the agenda for the ECR meeting and any decisions would apear in the formal report.

Mr Sylvester then discussed the 1958 Convention to be held in Chicago. He asked that any committees which did not originally plan to exhibit reconsider. He listed the following committees as having agreed to present exhibits: Resistance to Abrasion, Colorfastness of Textiles to Atmospheric Contaminants, Drycleaning Test Methods, Colorfastness to Crocking, Colorfastness to Wet and Dry Pressing, Flammability of Clothing Textiles, Damage Caused by Retained Chlorine, End-Usage Performance Tests, Identification of Finishes on Textiles, Dimensional Changes in Textile Fabrics, Evaluation of Fulling Agents, Nuclear Radiation, Wash and Wear, Water Resistance of Fabrics, Weathering of Textiles, Evaluation of Finishes for Resistance to Staining and Soiling, and possibly the Committee on Color.

A tentative program has been lined up for the Saturday morning symposium, the subject of which will be "AATCC Research at Work". Mr Sylvester will open the meeting at 9:40 am, calling on President Linberg for a brief message of welcome. Following this, Mr Dorn will discuss AATCC's contribution to industry through research. Thereafter, various committee members will speak, as follows: Jules Labarthe, chairman of the Committee on End-Usage Performance Tests; Joseph E Norton, chairman, Committee on Colorfastness to Light; Richard F Aurich, chairman, Committee on Damage Caused by Retained Chlorine; Albert E Johnson, chairman, Committee on Colorfastness to Drycleaning; Ralph B Smith, chairman, Committee on Colorfastness to Washing; George S Wham Jr, chairman, Committee on Wash and Wear; John F Warner, chairman, Committee on Dimensional Changes in Textile Fabrics. A discussion of developments in the evaluation of finishes for resistance to staining and soiling will also be included. Following a question-and-answer period, the TCR luncheon will take place from 12:30 to 2 pm. This will be open to the public, but admission will be by ticket only (to be purchased at the door).

Elliott Morrill, 1958 convention chairman, was called upon for a progress report. He stated that the theme of the Chicago Convention is geared to the research programs that have been going on throughout industry and particularly to the research program of AATCC.

There will be two programs on Thursday afternoon, October 30th. On Friday morning, October 31st, there will be one technical program. The Intersectional Contest will take place on Friday aftrnoon, and the banquet will be held on Friday evening. Mr Morrill stated his belief that the highlight of the Convention would be the "AATCC Research At Work" symposium on Saturday morning. He has been working closely with Albert Johnson on the corporate member relationship. Special mailings will go out to companies around Chicago, roughly within four hundred miles, to invite them to attend the Saturday morning meeting.

The Convention will officially close at the close of the TCR luncheon. It was reiterated that, there being so much to be gained for AATCC by this program, it should be opened up to interested people, essentially the public. Thus, the whole program, including the luncheon, will be open.

Mr Morrill thanked Mr Dorn and Mr Sylvester for the wonderful job they have done so far.

Mr Sylvester announced a new chairman for the Committee on Analytical Methods. P J Wood's chairmanship was terminated as a result of the three-year tenure rule and Emil G Wiest will be the new chairman.

J N Sivertson agreed to take over the chairmanship of the Committee on Water Absorbency. Charles Zimmerman, member of the Committee on Antibacterial Agents, was being replaced by Joanne M Spangler.

Six new candidates for membership on the Committee on Colorfastness to Light were announced as follows: Wilfred E Belanger of Chrysler Corp; G F Bush of G F Bush Associates; F H Leib, Chrysler Corp; Edwin L Grajeck; L Ricard, Arnold, Hoffman & Co, Inc; and Frederick T Simon, Union Carbide Chemicals Co.

George S Buck Jr of the National Cotton Council of America stated that, because of his new location in Memphis, Tenn, it would be difficult for him to attend TCR meetings. Mr Sylvester stated that he would submit Nelson F Getchell's name as a replacement for Mr Buck.

REPORT OF THE RESEARCH DIRECTOR———Harold W Stiegler, director of research, reported that there were four active projects in the AATCC laboratories. The projects are: Wear Abrasion, Colorfastness to Laundering Abrasion, Damage Due to Chlorine Retention, and Dimen-

sional Changes.

Accelerotor work for the Abrasion Committee is being carried on primarily in connection with the development of a rapid test for edge-wear abrasion in the Accelerotor, such as occurs on cuffs, folds, collars, and seams. The interlaboratory tests for reproducibility in the Accelerotor, which were run some time ago, will be summarized in exhibit form for the Chicago Convention and details will be published in the *Proceedings*.

It had been learned that the special rotor used in the Accelerotor which predicts colorfastness to laundering abrasion in the Accelerotor also has interesting application in dry wear testing. With many types of fabrics, more uniform abrasion is accomplished along with smoother running during the test and less reduction in efficiency of the abrasive liner.

Another step forward in wear abrasion testing in the Accelerotor is the standardization of the foam rubber liners which are placed under the abrasive liner to cushion the blow and provide for more even testing and smoother running in the instrument.

The joint committee of the AATCC-ASTM on Nonwoven Fabrics has started a project to apply the Accelerotor to abrasion problems in the nonwoven fabric field. Experimental work with nonwoven fabrics was carried out some years ago in the Accelerotor and provided a basis for more specific investigations. Recently a cross-section of nonwoven fabric types was submitted to the AATCC laboratories by the Committee to indicate the general nature of abrasion and wear in the Accelerotor on these modern types of nonwoven fabrics. Indications from preliminary work by the Committee were that evaluating abrasion resistance by weight loss of the specimen might provide worthwhile information. Reproducibility seemed to be quite possible. Visual examination of the specimens tested in the Accelerotor also provides a means of rating differences.

Dr Stiegler discussed the importance of alternate wear and wash cycle testing in the Accelerotor, whereby a short cycle of either grit or no-grit dry wear abrasion is carried out in the Accelerotor followed by a short cycle of wash abrasion in soap or synthetic detergent solution with the newly standardized white Hycar rubber-ribbed liner replacing the grit abrasive liner used in the previous dry cycle. These cycles may be varied in time from seconds to minutes, the net result being a more realistic testing of the fabrics with respect to weight loss by wear and color loss by laundering abrasion.

AATCC is in constant need of end-use results or effects on fabrics, particularly if field tests have been run on such fabric. Through the courtesy of P J Fynn, T F Cooke, chairman of the Committee on Abrasion, received some denims that have been tested for end-use in the field. This permits a study of the fibers and yarns and surfaces under the microscope and by other test means, to provide an end-use target toward which realistic Accelerotor test procedures may be arrived.

It was mentioned that between 110 and 115 Accelerotors were in use in the United States and foreign countries. In the Netherlands, there were 12; Japan 6; Canada 5; Germany 3; Denmark, Switzerland, and New Zealand had 1 each. In the United States, owners of multiple Accelerotors are Du Pont 6; American Cyanamid 5; Dan River Mills 2; Sears, Roebuck 2; Army QM 2; Monsanto 2; J P Stevens 2; universities, colleges and research organizations 20.

A new and interesting use of the Accelerotor is for testing "shedding," particularly for blankets, synthetic furs such as used for lining of coats, and so forth.

AATCC has received a Tension Presser from the US Testing Co on indefinite loan for the duration of the Committee on Dimensional Changes' investigations on shrinkage and restorability. Work was started on this instrument and on repairing and modifying the Dynamic Dimensional Change Meter. It is hoped that considerable information may be developed with these two instruments which would be helpful in understanding the response of fabrics to shrinkage and restorability tensions.

The Committee on Colorfastness to Washing has been very active in "industrial" laundering. Industrial laundering differs from commercial laundering in that much more severe conditions obtain in laundering heavily soiled work clothes where high temperatures, high alkalinity and considerable abrasion are necessary. An accelerated test for industrial laundering was desired by the Committee and developed in the AATCC laboratories by means of the Accelerotor. This rapid test, accomplished in close cooperation with Ralph Smith and his Committee, requires only a few minutes in the Accelerotor. It has been voted Tentative Test Method #87-1958. The Committee plans further work aimed towards extending the method to mixed fiber fabrics and synthetics (the present test is limited to cotton) where some dyes are not sensitive to abrasion but might change color in alkali or lose color in long soakings. It was shown sometime ago that such a situation may be coped with by means of a preliminary soak of one-half hour in the detergent solution before the specimen is subjected to a few minutes run in the Accelerotor. An interesting question had arisen as to whether synthetic fibers are being used to any extent in work clothing and whether or not such use might be increasing. By moving in on this problem, the Committee is preparing to provide an accessory method in anticipation of need.

The Committee on Damage Caused by Retained Chlorine has been very active with a broad program outlined. G M Kidder of the AATCC research staff made trips to a number of industrial laboratories to observe technicians using the present method in order to find out why certain discrepancies in results between laboratories occur. On the basis of these findings, the Committee will be in a better position to re-examine the method and make such changes as are necessary to insure interlaboratory reproducibility.

Dr Stiegler reported that the Japan-Reichhold Company had presented AATCC with its new Japan-Reichhold Wrinkle Tester free of charge. The instrument is well-designed and well-machined. It is being called to the attention of the Committee on Evaluating Crease Resistance for examination. This instrument and the Accelerotor for creating the washing phase of wash and wear evaluation within a few minutes, are used by the Japan-Reichhold Company and its customers in Japan for wash and wear evaluation according to a standard procedure. Multiple specimens may be passed through the Wrinkle Tester of angle measurement by an optical system. The Accelerotor wash tests require only a few minutes and obviate the need for long machine washing in order to follow crease-retention properties of fabrics and finishes.

SOILING OF CARPETS——J W Schappel, chairman of the Committee on Soiling of Carpets reported as follows:

"The primary objective of this Committee is to investigate service and accelerated soiling procedures for carpets with the aim of arriving at standardized procedures which will permit realistic appraisal of the soiling characteristics of carpets.

"Carpets comprising eight different fibers in natural color have been subjected to service and accelerated soil tests by six Committee members using procedures currently employed by the laboratories of the respective companies. Four members of the Committee are reviewing the findings of the soil tests to determine the interlaboratory correlation of the test procedures. Visual and instrumental methods of appraisal are being used to rate the soil levels of the samples.

"Tentative conclusions derived from the study of this Committee are:

1) A standardized visual evaluation procedure for soiled carpets is required. Pile lay direction and angle of viewing are important factors in examining carpets. The Committee suggests inspection by looking into pile at a 30° angle to the horizontal.
2) A jury ranking of a series of soiled samples proved good agreement between jury members, although marginal differences in soiling between carpets gave rise to some disagreement in ranking of samples.
3) Ranking of soiled samples by visual means yields insufficient information. Ranking fails to disclose soil level differences between samples. Therefore, methods which will afford a numerical gauging of the soil level are required.
4) Service soiling tests made at three different industrial locations proved similar soil behavior for the test fabrics. Rankings of the carpets soiled at the various locations were in good agreement.

"Future efforts will be directed towards the development of a gray scale applicable to carpet fabrics which will permit the grading of a carpet in its natural fiber color and irrespective of the initial presence of yellow and or gray color values. In addition, efforts will be made to standardize on a service soiling test and appraisal procedure."

WASH AND WEAR——G M Richardson reported for Dr Wham, chairman of the Wash and Wear Committee, who was unable to be present, on the proposed Tentative Test Method 88-1958. He discussed the test method for Wash and Wear properties of fabrics under the title "Appearance of Fabrics After Home Laundering," copies of which were distributed.

Dr Richardson spoke in part as follows:

"Our committee was organized in September of 1955 and since that time we have had about four or five rather formal interlaboratory tests, and as each test was run, we were able to focus in a little closer to better agreement. Today we are going to report on correlations of a test which we think is very good."

The Committee requested that TCR approve sending out the proposed Tentative Test Method to TCR members for vote by letter ballot as Tentative Standard. The motion was seconded and passed.

(The method appears on pages P578-P560 of the August 25th issue.)

EVALUATION OF FINISHES FOR RESISTANCE TO STAINING AND SOILING——The report of the Committee on Evaluation of Finishes for Resistance to Staining and Soiling was presented by H B Goldstein, chairman, as follows:

"The Committee now comprises 25 members who represent a wide cross section of the chemical industry, the textile finishing industry, and the consumer trade. The Committee met on May 21; fourteen members and three guests were present.

"Since the last meeting, an extensive bibliography of this subject and related fields was accumulated; copies of pertinent papers were distributed to the members.

"A review of this literature confirms the fact that adequate test methods are lacking to evaluate stain and soil resistance, especially when the staining material comprises oily or greasy material.

"In our meeting yesterday, a related topic was suggested for consideration by the Committee; namely, the problem of wet soiling during laundering. It was decided that this is a serious problem which is growing more serious daily, especially with regard to permanent finishes such as 'wash and wear', etc.

"After an extended discussion, it was decided that this was an appropriate topic for action by our committee. It is our understanding that no other AATCC Committee is now working on this problem.

"We decided that our committee would undertake work concurrently on the following two problems:

1) Develop a test method (or methods) to evaluate the resistance of finished fabrics toward staining and soiling by oily materials.
2) Develop a test method to evaluate the tendency of a fabric to stain or soil during laundering.

"I should emphasize that this last problem is not concerned with soil redeposition, which is a problem of detergency, but instead, we are concerned where a relatively clean fabric is laundered and it selectively picks up dirt so as to leave the washer grimier than when it entered.

"Two task groups were established to study these problems, and chairmen of these groups were appointed as follows: Hugh Bryce, chairman of the task group on resistance toward oily staining; and Charles Zimmerman, chairman of the task group on wet soiling.

"We hope to get work started promptly by both of these groups, and we expect to meet next in the Fall."

COLORFASTNESS TO WET AND DRY PRESSING——M W Goodell, chairman of the Committee on Colorfastness to Wet and Dry Pressing, reported as follows:

"The Committee has agreed that this area, which includes sublimation, will require at least three distinct tests. These are: 1) an accelerated aging test, 2) a steaming test, and 3) a pressing test. The initial effort is being made on the pressing test. It is felt that the scorch tester will provide a readily available test instrument with a satisfactory degree of accuracy. Fabrics have been collected and interlaboratory testing of the scorch tester vs hot irons will be started on these fabrics immediately. The same fabrics will be put through various finishing treatments, garment manufacturing steps and consumer-use conditions to determine the proper levels of temperature and time of exposure. It is planned that three temperature levels will be recommended as proposed in the ISO draft. However, rather than specifying the temperature for each fiber, the tests will be numbered 1, 2, and 3 in a manner analogous to the washfastness tests. Then, regardless of fiber or fiber blend, the specific test to be used can be picked on the basis of finishing and end-use requirements. It is expected that sufficient results will be available for an exhibit at the National Convention in October."

IDENTIFICATION OF FINISHES ON TEXTILES——Chairman B R Norwick reported as follows:

"The Committee on the Identification of Finishes in Textiles has been in existence for may years without publishing analytical methods, although individual members of the Committee have been extremely active. It is the opinion of the Committee

that sufficient progress has been made so that a preliminary presentation of the current approach is desirable. It must be borne in mind that many thick books have been and will be written on analysis methods, and that textile finishes embrace a tremendous and growing variety of materials.

"Textile finishes have been divided into eleven major classifications, and while a general method is presented, it is intended that each of the eleven classifications will be studied individually. At this time only two of the eleven are detailed. It is hoped that the remaining nine will be ready for publication in the future.

"I would like to comment on the excellent paper presented by the New York Section on the identification of dyestuff classes on dyed natural and synthetic fibers. In the paper as printed in the *American Dyestuff Reporter*, the statement is made that the procedure given makes it possible for a technician who is not experienced in the textile business to arrive readily at a correct conclusion. I regret to say that the methods which we have given will not enable a technician inexperienced in the textile business to arrive readily at a correct conclusion. I regret even more that the group publishing the paper on dyestuff analysis has seen fit to make its statement. There is an illusion even among chemists who are not directly engaged in analytical work that all analysis resembles qualitative inorganic analysis as taught in the freshman year of most colleges and the senior year of some high schools. This course has normally been taught in an extremely mechanical way and involves 25 possible metals and 20 possible nonmetals, all in solution. In actual practice, even inorganic analysis is far from being as simple as it is presented in such courses, and it is a dangerous and misleading thing for business executives to be encouraged to believe that they can obtain correct and quick information from untrained or low-grade technicians. It is this misconception on the part of a good many business men, especially in the textile industry, which has made them unable to obtain the major benefits to be derived from modern technology."

Discussion then ensued as to how to get comments and reactions on the proposals of the Committee from industry. It was suggested that the proposals be sent to the AATCC Headquarters for reproduction and mailing to TCR as a progress report, asking for comments and suggestions. It was agreed that this was to be done. Then later a test method could be drawn up and sent out for mail vote.

NUCLEAR RADIATION——A T Clifford, chairman of the Committee on Nuclear Radiation, presented the following report:

"The AATCC Committee on Nuclear Radiation was organized in 1956 by K H Barnard. The announced purpose of the Committee was to stimulate interest in the use of radiochemicals in the textile industry; that is, its activities were to be primarily educational. Since its organization, the Committee has cooperated closely with the Executive Committee on Research and with the Piedmont Section's Committee on Radiochemical Techniques. To simplify this report, the activities of the three committees in the field of radiochemistry will be considered together.

"In March, 1957, I A Berstein delivered a paper before the Piedmont Section entitled 'Applications of Radioactivity in the Textile Industry'. The paper was well received and won for Dr Berstein the seventh American Dyestuff Reporter Award as the best paper appearing in the *Proceeding* for that year. (ADR Vol 46, No. 11, P399, June 3, 1957).

"In its issue of October 21 (Vol 46, No. 21, P777), *American Dyestuff Reporter* published a report by the Committee on the subject 'Textile Users of Nuclear Radiation'. This report was prepared at the time Mr Bernard was chairman. Perhaps the most significant thing brought out in this report was that the textile users of radiochemicals were mostly companies concerned with textiles only indirectly. Thus, almost every firm engaged in coating textile fabrics was using beta gauges, but not a single firm engaged exclusively in the manufacture and finishing of textile fabrics.

"In March, 1957, on the recommendation of the Executive Committee on Research, the Council appropriated $2000, and the Piedmont Section $500, to North Carolina State College to help pay for a preliminary investigation which would serve as a guide for further work. North Carolina State will make a formal report on this shortly. In an informal report to the Committee, H A Rutherford, director or the North Carolina State project, has revealed that they are getting some exceedingly interesting results in their work with the beta gauge. For one thing, they have found that this instrument will do exactly what was predicted; that is, it will measure the wet pickup on padded fabrics with a high degree of precision.

"During the current year the Committee has continued its educational activities with special emphasis on getting radiochemical laboratories established in textile plants. At present most of the work with radiochemicals in textiles is being done at institutions such as MIT, TRI, ITT, North Carolina State, Clemson College, Oak Ridge Institute, and Southern Regional Research Laboratory. Georgia Tech will soon have a radiochemical laboratory. The Commitee feels that much of the excellent work going on at these institutions cannot be reduced to practice until the textile plants themselves become actively interested. The Committee is cooperating with the institutional laboratories but its main efforts are now being directed toward private textile plants. Recently, the Committee submitted a report (written by Dr Berstein) designed to correct some of the common misconceptions which have arisen among those responsible for the smaller textile laboratories. This report, *American Dyestuff Reporter* Vol 46, P297 (1958), discusses costs, the training of personnel, safety requirments, licensing procedures, etc.

"In March, 1958, the Committee on Nuclear Radiation cooperated with the School of Textiles, Clemson College, in a Seminar on the Utilization of Radioisotopes in the Textile Industry. Other cooperating agencies were the United States Atomic Energy Commission, the Southern Regional Advisory Council on Nuclear Energy, and the South Carolina Textile Manufacturers Association.

"The AATCC was well represented at the Seminar. More than half of the guests were AATCC members; the rest were mostly executives representing the South Carolina Textile Manufacturers Association. The Committee on Nuclear Radiation had an active part on the program. W T Rainey was on the planning committee and was chairman of the program committee. A T Clifford was a member of the planning committee and he presided at one of the technical sessions. I A Berstein, O K Neville, W T Rainey, and H J White gave papers. As a matter of fact, Berstein's paper was an elaboration of the Committee report mentioned above.

"The Committee believes that its efforts, and those of other groups interested in the same problems, are beginning to get results. The chances are that, in the near future, several textile plants will establish their own

radiochemical laboratories. The Committee hopes then to set up some cooperative research projects similar to those now being conducted by other AATCC research committees.

"The Committee took advantage of the Clemson Seminar to hold its spring meeting with nine of the eleven members present. The business transacted at this meeting may be summarized as follows:

Three new members were elected: O K Neville, Nuclear Chicago Corp, Chicago 10, Ill; F M Sell, Deering Milliken Research Laboratories, Pendleton, SC; and J C Arthur, Southern Regional Laboratory, USDA, New Orleans 19, La.

A special effort will be made to enlist members from those Sections not now represented.

The Committee is trying to comply with the request of the Executive Committee on Research to enlist Southerners not actively engaged in AATCC committee work. It is doing this, however, without in any way compromising its position as a national committee.

The Committee will continue its regular yearly meetings at the National Convention; it will hold other meetings from time to time, not necessarily in conjunction with the meetings of the Technical Committee on Research, but wherever it can get the best attendance.

The Committee is planning a live exhibit for the Chicago Convention which will include:

A cloud chamber showing vapor trails caused by emanation from radioisotopes.

A demonstration of the use of beta gauges for measuring the thickness of textile coatings and the wet pickup in padding operations.

A demonstration of the use of radioisotopes in analytical procedures.

A demonstration showing the use of radiochemical tracers in textile research.

Counting devices—audience participation showing safe radioactivity in radium-coated wrist watches.

Photographs showing radiochemical laboratories at Clemson College, Southern Regional Laboratory, N C State, and perhaps one or two other institutions.

Reprints of bibliographies, technical papers, and committee reports."

DIMENSIONAL CHANGES IN TEXTILE FABRICS——Albert E Johnson reported for the Committee on Dimensional Changes in Textile Fabrics as follows:

"The Committee approved in principle, subject to minor editorial corrections, a test for Dimensional Changes in Woven Tested Fabrics (Excluding Wool). Upon final approval by the Committee, the test will be submitted to TCR for letter ballot. The test provides for shrinkage determinations at four levels of launderability from hand laundering to working at the boil. The Committee also moved to adopt the present ASTM test for shrinkage of knitted fabrics to these same levels of launderability with a view to providing a similar AATCC test for dimensional changes in knit goods."

Approval was granted by TCR for submission of the test method "Dimensional Changes in Woven Textile Fabrics (Excluding Wool)" to letter ballot of TCR.

END-USAGE PERFORMANCE TESTS——This Committee approved a standard form for recording textile serviceability complaints in line with the objectives of the interlaboratory statistical plan approved by TCR last January. The forms are subject to some minor editorial changes to be approved by the Committee at an early date and thereafter will be submitted to TCR for letter ballot. The Committee is developing a manual on the use of these forms as an aid to laboratories and to assure accuracy of the information. No decision has been made as to how laboratories will be asked to submit reports on these forms for periodic tabulation analysis and distribution by AATCC Headquarters to members. In all probability, however, the tabulations will not be more often than six months. All laboratories will be invited to cooperate in the plan.

After some discussion, approval was granted by TCR to submit the forms to letter ballot.

COLORFASTNESS TO WASHING——Mr Mandikos read the report of the Committee on Colorfastness to Washing as follows:

"The Committee will need approximately 25 feet of space for the exhibit at the Chicago Convention which will include:

Exhibits of the 2A, 3A, and 4A Tests.

Comparison of old 2, 3, and 4 Tests vs 2A, 3A, and 4A.

Launder-Ometer with both metal cylinders and glass jars.

Exhibits for Industrial Laundering Test Method 87-1958 to include 1) two cotton fabrics, and 2) five mixed fiber fabrics.

Accelerotor.

"Ralph Smith will prepare a write-up of the New Industrial Laundering Test, Tentative Method 87-1958, using the Accelerotor, for publication in an early issue of the *American Dyestuff Reporter*.

"The Industrial Laundering test has been approved for cotton fabrics only, and work is now under way at Lowell to extend the method for mixed-fiber fabrics. This work has shown that a 30-minute preliminary boil with a nonionic and alkali, followed by the three-minute Accelerotor test specified in method 87-1958 is necessary to duplicate the average of five industrial launderings." (Mr Mandikos showed exhibits on five mixed fiber fabrics).

"It is now planned to run an interlaboratory survey on the five mixed-fiber fabrics and at least one cotton fabric, all to be run employing the preliminary boil.

"Following the interlaboratory survey and checking of results, the test method will be rewritten to include the preliminary boil with a footnote to the effect that the preliminary boil may be eliminated if it is known that the dyes do not bleed or that any protective finish present is not removed by an alkaline boil."

ANTIBACTERIAL AGENTS——H E Wilde, secretary, reported as follows for the Committee on Antibacterial Agents:

"The most recent meeting of this committee was held May 9 in the Dept of Agriculture Building in Washington, D C.

"All of the members were present, and two visitors from the Dept of Agriculture, Dr McNeil and L S Stewart, sat in on the discussion.

"The status of the Committee's activities from the time of its inception to date were read so as to bring all of the present members up to date on what had been done in developing these test methods.

"It was brought forth that this was not a new test method or a 'brain child' of any one individual, but the old FDA 198 method that was last published in 1931, with several refinements so as to make it applicable to the testing of textile materials.

"Dr Reddish in his work used this test for testing of pharmaceuticals and medicinals where halos from two mm to nine mm are sought. In circular 198, no mention is made as to the

size of halos but it states that a halo denotes defusion.

"This method for the testing of textiles calls for the condition of the plate in the contact area, with or without a halo. The method also states that it is not applicable to all types of materials and that other test methods must be investigated.

"It was pointed out that a 'Tentative Test Method' was a necessity. There are so many companies interested in the development and use of bacteriastatic compounds that there is considerable confusion as to how to test either the compounds or the treated fabrics.

"It has been proven that this method, if followed precisely as written, is reproducible.

"Ratings are based on the following classifications: complete inhibition in the contact area, partial inhibition or no inhibition. Any halo or zone of inhibition surrounding the test specimen to be reported separately.

"The members agreed to submit this method as a 'Tentative Test Method' allowing for any minor changes that might be made in the future."

Mr Wilde then asked for approval of TCR to submit the method to letter ballot to TCR. The request was granted.

COLORFASTNESS TO LIGHT——J E Norton, chairman, reported as follows for the Committee on Colorfastness to Light:

"The Lightfastness Committee met at 9:00 AM Thursday morning. Four task groups under this Committee held meetings on Wednesday and reported on their activity.

"Group I, under C H A Schmitt, is continuing the daylight exposure program for 12 successive months at three locations. This program will be completed in July of this year. Samples as they are completed are being mounted and evaluated. After the test program is completed the results will be analyzed and reported.

"Group II, under the chairmanship of V L Chase, held its first meeting. Work on correlating and improving laboratory test methods with sunlight and daylight exposured will be shortly turned over to the group as a basis for further work of the group.

"Group III, under the chairmanship of V S Salvin, will explore the effect of various astmospheric contaminants in combination with light. After some preliminary investigations, it is quite probable that we will wish through channels to have some work done at Lowell. Dr Salvin has prepared a short paper on the preliminary work done by his group and this is being forwarded through channels for publication. It is hoped that publication of this paper will bring a recognization of the problem and stimulate interest in work of the Committee." (Paper appeared in *American Dyestuff Reporter* 47, No. 13, P450-1, June 30, 1958).

"Group IV, under the chairmanship of E L Grajeck, held an organizational meeting. This group will investigate the desirability and possibility of developing methods for evaluating colorfastness to weathering.

"Results of the letter ballot on submitting our daylight and carbon arc methods to the ISO received 84 affirmative and no negative votes. Editorial comments were received from two people and the suggested corrections have been made."

WATER RESISTANCE OF FABRICS——R K Worner's report for the Committee on Water Resistance of Fabrics was read by Mr Mandikos as follows:

"A general meeting of the Committee on Water Resistance of Fabrics has not been held since the new chairman took office on January 1. However, a small planning group met in Washington, D C on March 19 in conjunction with the meeting of Committee D-13, ASTM Task Group on Water Resistance of Fabrics. Those present were Carlo DeMarco, George A Slowinske, Harold W Stiegler, and Ruby K Worner. This is a report of that meeting. Part I is concerned with the status of ASTM test methods as they are related to those of the AATCC. Part II deals with studies in progress or planned by the AATCC Committee.

"Part I.—Acting as members of the ASTM Task Group, the draft of the AATCC alternate Dynamic Absorption Test, which had been written up in appropriate format for addition to ASTM D 583-54, was reviewed and edited so as to be satisfactory for presentation to Subcommittee B-1. If the method is approved by letter ballot, the ASTM Task Group will be dismissed.

"Subcommittee B-1, 'Chemical and Performance Test Methods' has been considering deletion of D 583-54 from the manual of 'ASTM Standards on Textiles' since most of the tests in it have been developed by the AATCC Committee and most of the members interested in it are also members of the AATCC. However, there are three important test procedures in D 583-54 which are not covered at all or are merely mentioned in the *Year Book*,—namely, the Drop Penetration Test, which is also given in Method 5520 of Federal Specification CCC-T-191b, is not included. The Suter hydrostatic tester, (also in Federal Specification CCC-T-191b, Methods 5514 and 5516) is mentioned in a Footnote in Method 18-1952 where it is stated that the Suter apparatus has been approved as an alternate or optional instrument for use in place of the AATCC hydrostatic tester.

This method covers only determination of the hydrostatic head at which leakage occurs, but does not include a procedure for determining the amount of leakage in a given time at a fixed head. Because of these additional test procedures in ASTM D 583-54, its retention with addition of the alternate dynamic absorption test (subject to letter ballot) was approved for the time being. It was suggested, however, that cooperation should be established with the AATCC in order to unify the methods of the two organizations.

"Part II.—The work of the AATCC Committee on Alternate Dynamic Absorption Test (ie, using the metal tubes in the Launder-Ohmeter in place of the tumble jar) is essentially completed except for some further checks of the behavior of laundered fabrics in the test. Mr Slowinske has agreed to follow through on this work.

"As new business, a study of blotting papers, particularly with respect to absorbency, was proposed by Mr De Marco. There are several aspects to the problem that make such a study a timely one. In the AATCC methods, two qualities of paper are required: namely, the "White AATCC Textile Blotting Paper," prescribed in Standard Methods 21-1957, 35, 1952, and 42-1957 (static immersion absorption, rain, and impact penetration tests respectively) — the supply of which is limited — and the blotters specified in Note 4 of Tentative Method 70-1952 for the dynamic absorption test with the tumble jar, for which three sources of supply are given. At present, the AATCC specification has different absorbency requirements than are given in the corresponding Federal Specification and ASTM methods, but consideration in being given to changing the requirement in the Federal Specification (which presently agrees with the ASTM) to that specified by the AATCC. Both the AATCC and Federal Specification methods refer to Federal Specification No. UU-P-31b,

for paper, General Specifications and Methods, for the absorbency test. These methods permit indiscriminate use of pipettes without regard to rate of delivery. Consequently, various absorption times could result when pipettes of different rates are used. For these reasons, and since the AATCC committee on water resistance is currently undertaking the development of an alternate dynamic absorption method for which blotters are required, it was decided to conduct a general review of the subject of blotting papers used in water resistance tests. The investigation will include not only a study of absorbency and its measurement, but also of other properties such as weight, thickness and bursting strength, particularly as they affect the end result. Mr DeMarco has agreed to be chairman of a small task group on blotting paper to study the problems involved."

TEST METHODS FOR MERCERIZATION——— S M Edelstein, chairman, reported for the Committee on Test Methods for Mercerization as follows:

"Just a brief bit of informal discussion for background. This report of the Committee is the result of the fact that some 25 years ago the Research Committee of the Association was more or less informal. Some 23 years ago or so, there was a Research Committee consisting of men from the South, working on mercerization. One of the first developments of this Committee was a method for determining the presence of mercerization and the method involved the determination of various numbers. This method was presented simply as a paper in *Dyestuff Reporter*, without any official standing or status in the Association. This method has been used, I would say, internationally, for some 20 years or so. It has generally been accepted, but there were no standard methods for the details. Many people took upon themselves the modification of the test which caused trouble, and there has been disagreement, unless the original method was followed.

"The present Committee has been concerned simply with taking steps to make this method an official part of the Research Committee's methods. The Committee consists of Edward W Lawrence, of Chanston Print Works Co; Paul B Stam of J P Stevens Co, and myself. The work of this Committee has been concerned with rechecking the (barium) numbered test for mercerization. A detailed method has been written. Different samples of mercerized goods have been prepared by Cranston Print Works Co and the test method has been used by the three laboratories that were interested, on these fabrics.

"Extremely close check results have been obtained by all three laboratories and the method, as written up by this Committee, is recommended as a tentative method for the Association. The Committee would like to enlarge itself and to continue work for the moment on the (barium) number test for mercerization on resin-treated goods.

"Now we have a method written up that is ready to be submitted, with all the reports from the laboratories."

A motion was made, seconded and passed that the test method be submitted to vote of TCR as a Tentative Standard.

GUEST SPEAKER ——— A paper was given by George Switlyk, statistician, Engineering Department, E I duPont de Nemours & Co, Inc, on the subject "Statistical Approach in Developing Test Methods". (The full test of Mr Switlyk's paper appears on pages P628-30 of this issue of *American Dyestuff Reporter*).

At the conclusion of the paper, Mr Sylvester stated that he would like to establish an "Advisory Committee on the Use Of Statistical Methods." A motion was made, seconded, and passed that such a committee be set up.

The session adjourned at 4:30 PM.

Respectfully submitted
H W Stiegler
Secretary

Membership Applications

SENIOR

Thomas J Cronin Jr—Mgr of new product development, textiles, Quaker Chemical Products Corp, Conshohocken, Pa. Sponsors: J H Davids, J B Irvine. (DV).

James E Garrett—Finishing & bleaching supt, Opelika Mfg Co, Hawkinsville, Ga. Sponsors: J M Cook, C R Smith. (SE).

Robert C Hargreaves—Technical sales (textile chemicals), Quaker Chemical Products Corp, Conshohocken, Pa. Sponsors: J H Davids, J B Irvine. (DV).

William J Maher Jr—Textile engineer, Puritan Looms, Inc, Philadelphia, Pa. Sponsors: P Theel, W F Prien. (DV).

Werner F Schulz—Sales technician, Ciba Co, Inc, New York, NY. Sponsors: S N Glarum, P J Luck. (Met).

George J Western—Technical sales & service, A J Lynch & Co, Los Angeles, Calif. Sponsors: F H Ahl Jr, J R Nehmens. (PSW).

Arthur Haavie—Overseer of finishing, Cedartown Textiles, Uxbridge Worsted, Cedartown, Ga. Sponsors: H W Berry, W B Griffin. (SE).

ASSOCIATE

Richard E McSweeney—Head, Merchandising Dept, (formerly dyestuffs buyer), Geigy Chemical Corp, Yonkers, NY. (Met).

Martha J Ulrich—Extension clothing specialist, Agricultural Extension Service, Univ of Wyoming, Laramie, Wyo. (PNW).

Elected to Membership

AUGUST 14, 1958

SENIOR

Donald R Baer — Tokiharu Narita
Claude E Bozeman — Fernando B Olvera
H Haley Ector — Barney E Sampson
Ralph M Greene

ASSOCIATE

Etta L Finley — Marion A Pool
Duck Soo Kim — Margaret L Ross
Alice C Linn — Brannon H Wilder

STUDENT

Miguel S Marina — Armando Munoz
Shirish B Mehta

TRANSFER TO SENIOR MEMBERSHIP

John D Egan Jr — Edward L Kellev
Adeline M Hoffman — Betty Faye Smith

ORGANIZATION OF THE AATCC

FOUNDER

LOUIS A OLNEY
(deceased)

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STUDENT CHAPTERS

Alabama Polytechnic Institute, Bradford Durfee College of Technology, Clemson College, Georgia Institute of Technology, Lowell Technological Institute, New Bedford Institute of Technology, North Carolina State College, Philadelphia Textile Institute, Rhode Island School of Design

News of the Trade

W&W Developments to Highlight 7th Chemical Finishing Conference

Recent developments in wash and wear finishes for white cottons will be the subject of one full session at the seventh annual Chemical Conference to be held at the Statler Hotel in Washington on October 1 and 2.

At the session, papers will be presented covering improvements in formulations for white cottons now in commercial use and in pilot-plant stages. Research progress in these bleach-resistant finishes has led to the market success of cotton wash and wear dress shirts during the past year, and also to the introduction of "no-iron" cotton sheets and pillow cases within the past few months.

Further developments of the types to be reported at this conference may be of major importance in cotton's competition with synthetics for future wash and wear markets.

Rosser L Wayland Jr, Dan River Mills, Danville, Va, will compare improved triazone resins, the type used in a high percentage of white cottons today, with other commonly used resins.

Improved finishes based on blends of epoxy resins with different resin bases will be discussed in a paper by D D Gagliardi and F B Shippee, Gagliardi Research Corp, East Greenwich, RI.

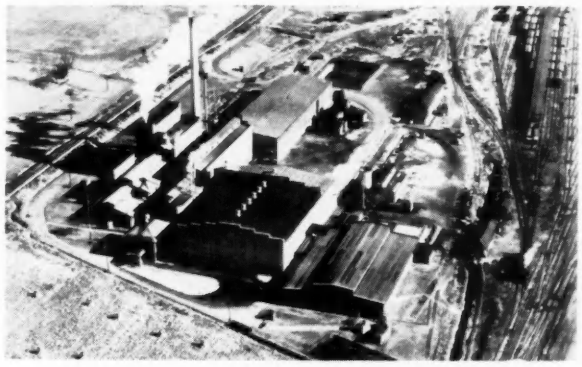
Acetals, another nonchlorine-retentive wash and wear treatment, will be covered in a presentation by James B Irvine, Quaker Chemical Product Corp, Conshohocken, Pa.

Properties of a new resin formulation still in test stages will be disclosed by Theodore F Cooke, American Cyanamid Co, Bound Brook, NJ. Laboratory tests on this new product will be covered as will commercial use tests, including high-temperature washing and commercial laundering.

The conference, sponsored by the National Cotton Council of America, is expected to be attended by more than 300 textile scientists from private industry, government and educational institutions.

Huisking Acquires Glyco

Chas L Huisking & Co, Inc, a leading merchandiser and manufacturer of raw materials for the pharmaceutical industries, has acquired all assets of Glyco Products Co, Inc, producers of chemicals for the textile and other industries. The transfer took place August 22nd.



Williamsport, Pa, plant of Glyco Chemicals Corp

According to a recent announcement issued by Charles L Huisking, founder and president of Chas L Huisking & Co, Inc, 417 Fifth Avenue, New York, NY, and Harry Bennett, president and Edward Rosendahl, executive vice president of Glyco Products Co, Inc, 350 Fifth Avenue, New York, NY. Huisking will form a new corporation, Glyco Chemicals Corp, as a manufacturing and sales subsidiary.

In making the announcement, Mr Huisking, who founded the Chas L Huisking Co in 1910, noted that the day of the chemical merchant is declining. "There is a tendency", he said, "for the consumer to go directly to the primary producing manufacturer for his needs. In line with this trend, Huisking in acquiring Glyco is taking the necessary steps to expand its research and manufacturing position, broaden its markets and serve the needs of its customers more efficiently."

Chas L Huisking & Co, Inc, one of the great merchant chemical companies, markets drugs, chemicals, alkaloids, oils and vitamins and operates the Clintbrook Chemical Co, Inc in Lyndhurst, NJ. Its new subsidiary, Glyco Chemicals Corp, will continue to produce and market synthetic waxes and emulsifiers for the textile, adhesive, surface coating, plastic, metal, and rubber industries, as well as emulsifiers and stabilizers for the food industry. It also produces chelating agents, the Tetrines.

Within the past few years it has greatly expanded its production of DMH, the key intermediate in the production of several novel chemical products of rapidly expanding importance.

According to Mr Huisking, officers of Glyco Chemical Corporation will include William W Huisking (now vice president, Chas L Huisking & Co, Inc) as president, himself as chairman of the board. Mr Bennett and Mr Rosendahl are retiring from the business but will be available for consultation. Eugene McCauliff, vice president in charge of sales and development, W H Roberts, vice president in charge of operations, and M S Green, vice president, finance, will continue to serve in these capacities with the new Glyco Chemicals Corp.

The newly formed Glyco Chemicals Corporation will continue to operate its Williamsport, Pa, plant. This includes 150,000 sq ft of manufacturing facilities over 40 acres of land. The plant employs 100 people in the Williamsport area.

Houghton Buys New Plant Property in Georgia

An industrial property on a five-acre plot in Carrollton, Ga, near Atlanta, has been acquired by E F Houghton & Co, Philadelphia.

Buildings will be modernized and enlarged for use as a manufacturing and warehousing facility, with equipment to be installed for blending, compounding and treating industrial oils and chemicals for textile, paper, and metal processing. Location of this new plant in the heart of Georgia is designed to speed up deliveries and provide convenient stocks.

Houghton also maintains plants in Detroit, Chicago, San Francisco, Minneapolis and Toronto, Canada, in addition to its main plant and laboratories in Philadelphia.

Commercial Production of Creslan to Begin

Commercial production of American Cyanamid Company's Creslan acrylic fiber will begin late this year at one of the world's most modern and efficient fiber production facilities —the company's 25 - million - dollar Santa Rosa Plant near Pensacola, Fla.

Construction of all major installations at the plant is now virtually complete, according to Aldon Loosli, general manager of Cyanamid's Fibers Division, and the installation of processing equipment is proceeding. Initial shipments of acrylonitrile and other Creslan raw materials have been received, he said, and key members of the plant's operating staff are now on duty preparing for its start-up.

After production begins in late fall, Mr Loosli asserted, output at the huge plant will be gradually increased until the plant reaches its full annual capacity of 27 million pounds of Creslan staple and tow.

Limited introductory marketing of Creslan to the textile trade, which is to begin this fall, will be from the output of Cyanamid's fiber pilot plant, located at the company's research laboratories in Stamford, Conn, he noted.

The Santa Rosa Plant, located on the eastern shores of Escambia Bay about 20 miles from Pensacola, is on a direct line of supply from Cyanamid's source of acrylonitrile, principal raw material of Creslan, to the leading textile centers. The company's Fortier Plant, near New Orleans, is the nation's largest production facility for acrylonitrile—a raw material commercially pioneered in the United States by Cyanamid.

Begun last summer, the Santa Rosa Plant will employ about 425 persons upon completion. More than 1,000 are currently employed in its construction.

The plant reportedly is one of the world's most versatile and efficient facilities of its type, embodying the newest concepts in fiber production. In the plant's main manufacturing building, one fifth of a mile in length, processing equipment is laid out to permit close control of production and the greatest efficiency in effecting change-overs in denier, brightness and other fiber specifications.

The flexibility of the Santa Rosa Plant is in keeping with the great versatility of Creslan as a fiber, Mr Loosli said. The well-rounded marketing program which has been developed for Creslan requires a plant which can meet the swiftly changing needs of dozens of fashion markets without lengthy shutdowns or sharp drops in production, he explained. The Santa Rosa Plant, it is claimed, will be able to meet all the varying needs of the apparel and home furnishings trade without sacrificing capacity.

Coincident with the construction of the Florida plant, Cyanamid has conducted exhaustive product development and wear-test programs for Creslan in more than a score of end-use areas. These tests, Mr Loosli asserted, will enable Creslan to reach the consumer next spring with an unsurpassed record of wear-testing prior to plant production.

Mr Loosli noted that Cyanamid has established headquarters for the Fibers Division in the newly completed office building at 111 West 40th St, New York, NY, to provide better service to the textile industry. Sales, merchandising, advertising, promotion, technical service and administrative offices, as well as full-scale display facilities for Creslan, occupy the building's entire 16th floor.

New York Chapter, AIC, Program Set

James M Crowe, executive editor of the American Chemical Society Applied Publications, will survey job opportunities open to the aggressive chemist of any age for New York Chapter members of the American Institute of Chemists at their first fall meeting. The address, entitled "The Best Job for You", will be given at 7:30 pm in the Bogert Room of the Chemist's Club on Thursday, September 25. The room will be opened at 5:30 for a social hour and dinner will be served at 6:30.

Officers of the Chapter have built this year's program around the theme, "Achieving Professional Success on a Do-It-Yourself Basis."

Following Mr Crowe's lead-off, Marlin G Geiger on November 20 will discuss ways and means of achieving success in the job selected. Mr Geiger holds the position of executive vice president at W R Grace & Co.

On February 6, Wallace R Brode, scientific advisor to the Department of State, will uphold the thesis that the chemist should contribute actively to government policy.

The science editor of the *New York Herald Tribune*, Earl Ubell, will present his ideas April 16 on how the chemist can achieve community recognition.

In addition to the regular program, an Honorary Membership award will be made January 8 and an Honor Scroll award May 28 at the Hotel Shelburne.

Parks Heads New Textile Consulting Labs

American Research and Testing Laboratories, Inc, a new consulting organization specializing in textile problems, has been established in Pawtucket, RI. W George Parks is president of the firm and director of the laboratories, which are equipped to handle a broad range of chemical and physical evaluations of textile fibers and fabrics, including studies on fire-resistance, construction of fabrics which have special properties, and testing textile materials to ensure that they meet government purchasing specifications.

Dr Parks is head, Department of Chemistry, Univ of Rhode Island, and chairman of the University's Committee on Research.

Midland Coatings Society to Open 3rd Season

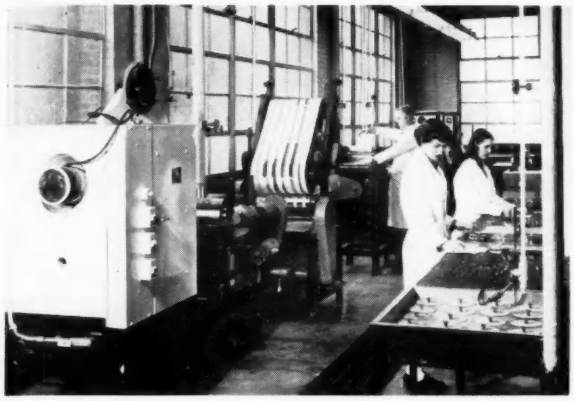
Turner Alfrey Jr will present a talk entitled "The Relation Between Molecular Structure and Polymer Properties" to headline the opening of the third season for the Midland Coatings Society on Tuesday, September 23rd. The meeting will be held in the Research Auditorium, Dow Library, Midland, Mich, starting at 7:30 PM. Dr Alfrey is director of the Polymer Research Laboratory of the Dow Chemical Company.

The program for the evening will include the official installation of officers for the 1958-1959 season. Officers for the coming year are: M C Carpenter, *chairman;* D P Sheetz, *vice chairman;* R C Hedlund, *treasurer;* and J F Vitkuske, *secretary.*

Werner Textile Consultants to Move N Y Office

Werner Textile Consultants, one of the leading consulting firms in its field, has signed a long-term lease for the relocation and enlargement of its New York office. The move to the new quarters, formerly occupied by the Finishing Division of Cone Mills in the Lowenstein Building at 1430 Broadway, will take place this month.

For the past 18 years Werner Textile Consultants, with offices in New York, Larchmont, NY, and Atlanta, Ga, had maintained its New York office at the Lincoln Building, 60 East 42nd Street.



Testing and reporting services rendered under the Eastman Plan for Specification Dyeing are carried out in Eastman Chemical Products dye service laboratories in Lodi, NJ, and Kingsport, Tenn.

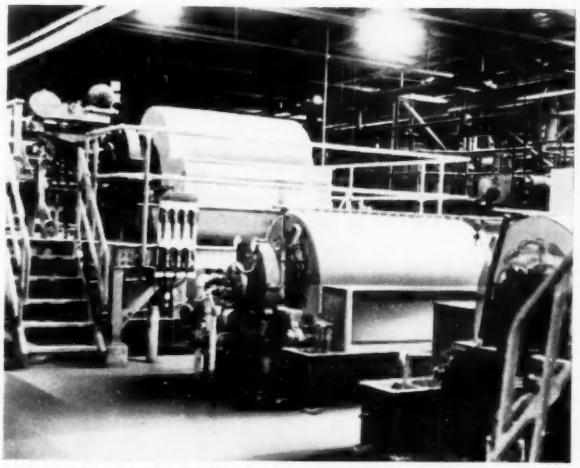
Eastman Plan for Specification Dyeing

As a result of a recent study into the problems of colorfastness control in the textile industry, Eastman Chemical Products, Inc has announced a new dye service plan designed to aid dyers, converters and manufacturers in establishing and maintaining standards of colorfastness for the piece-dyed acetate fabrics they handle.

Key to the Plan, called the Eastman Plan for Specification Dyeing, is a comprehensive testing service to be carried out at the company's dye service laboratories in Lodi, NJ, and Kingsport, Tenn. Under the Plan, converters and manufacturers are encouraged to specify the precise degree of colorfastness required for their particular markets in terms of the number of gas cycles, Fade-Ometer hours, or other standard test methods established by AATCC. These minimum requirements may apply to a given quality of fabric, or for an entire line if used in a single trade.

To achieve the control provided by the Plan, the dyer and the converter must first reach an agreement regarding minimum fastness requirements. At this point the dyer arranges with Eastman for the necessary testing service and, as each lot of fabric is dyed, forwards a sample of the fabric to the nearest Eastman dye service laboratory where the specific tests are performed. A complete report is furnished to the dyer or, at the dyer's request, to the converter including test swatches, dye lot numbers, and all other necessary identifying information. This test report becomes the basis for the converter approving shipment of the fabric lot or acknowledging its acceptance.

Backing up the testing and reporting service rendered under the Eastman Plan is the use of Eastman dyes.



URANIUM PRODUCTION GETS ASSIST FROM MAN-MADE FIBER———Acid-resistant Dynel filter fabrics reportedly provide many times the useful performance life with substantial cost savings in the refining of uranium ore. Woven of Union Carbide's acrylic fiber, the cloths are used on mammoth Eimco drum and precoat filter units operating 24 hours a day to withstand deterioration from sulfuric acid solution in which the ore is processed and continuous abrasive action from scraping and vacuum blow cycles. This means increased production efficiency by eliminating frequent stoppages to replace worn filters.

Work Begins on New USDA Wool Processing Laboratory

Construction of a new wool-processing laboratory, a unique facility in the U S Department of Agriculture and the country, has begun on the grounds of the Western Utilization Research Laboratory in Albany, Calif.

The new building will be two stories high and have 18,000 square feet of floor area. It will permit a large expansion of the Department's research on new wool products and processes. Pilot-scale equipment for processing raw wool into finished worsted fabric will be installed. Later the plant is to be equipped also for production of knitted and soft woolen goods.

Research on improved methods of processing wool, new wool products, and treatments to make wool garments easy to care for will be conducted in the new building, it is reported. Improved wool properties sought include resistance to yellowing and shrinkage, permanent pleats, and more rapid drying, and less need for pressing.

The new plant is expected to be in operation early in 1959. It is an addition to the Wool and Mohair Laboratory, part of the Western Utilization Research and Development Division of USDA's Agricultural Research Service. In charge of the new processing facility will be Amon H Brown. His staff will consist initially of 15 scientists, engineers, and technologists. Michael J Copley is director of the Division, and Harold P Lundgren is chief of the Wool and Mohair Laboratory.

Conference on Military Applications of Fabrics for Coating

Under the sponsorship of The Textile Fabrics Committee, Quartermaster Advisory Board, National Research Council, National Academy of Sciences, a Conference on Military Applications of Fabrics for Coating will be held October 16-17 at the Quartermaster Research and Engineering Ctr, Natick, Mass.

Topics to be covered at the Conference will include: "Military Applications for Coated Fabrics"—S J Kennedy, QM Research & Engineering Ctr, R Grubb, Philadelphia Naval Shipyard, and J H Ross, Wright Air Development Ctr, USAF; "Design for Climatic Extremes—Austin Henschel, QM Research & Engineering Ctr; "Interaction of Fabric Structure and Coating Type as Affecting Performance"—M M Platt, Fabric Research Laboratories, Inc; "Fabrics for Coating"—W Corey, The Landers Corp; "New Fibers and Yarns for Coated Fabrics"—R C Pike, Textile Fibers Dept, The Du Pont Co; "Technical Advancement in Coating Materials for Fabrics"—F H Fritz, Elastomer Chemical Dept, The Du Pont Co; "Evaluation of End-Use Performance"—K L Keene, U S Rubber Co; "Coating Technique"—speaker to be named.

Further information may be obtained from W George Parks, QM Advisory Board, National Research Council, National Academy of Sciences, Washington, DC.

Process Chemicals Dept Formed by Cyanamid's Industrial Chemicals Div

Formation of a Process Chemicals Department by the Industrial Chemicals Division of American Cyanamid Company has been announced by G W Russell, division general manager.

The new department, created through the merger of the company's Heavy Chemicals and Manufacturers Chemicals Departments, will service chemical needs with an extensive line of intermediate, heavy and specialty chemicals for the process industries.

Among the chemicals that will be handled by the new department are cyanamide and its derivatives—dicyandiamide and melamine—alum and acids, phthalic and maleic anhydride, surface-active agents, cyanuric chloride and a wide range of other specialty chemicals.

H C Milton has been appointed manager of the department and R M Goddard has been named sales manager. Prior to assuming his new post, Mr Milton served as manager of the Manufacturers Chemicals Department. Mr Goddard was assistant manager of the Heavy Chemicals Department. Both men began their service with Cyanamid over 20 years ago.

Principal office and eastern regional headquarters of the Process Chemicals Department will be in New York. In addition, there will be a western regional headquarters in Chicago as well as sales offices in 17 key cities throughout the country.

NYPC Announces Fall Schedule

The New York Pigment Club, now in its fifth year, will hold its first meeting of the new season on Friday, September 11, at which Joseph J Quinn Jr, Bowen Engineering, Inc will discuss "Adapting the Spray Drying Process to Pigment Manufacture".

The Club holds all its regular meetings at Fraunces Tavern, Broad and Pearl Streets, New York, NY, at 6:30 PM. For dinner reservations, contact Robert Copland, secretary, L & R Organic Products, 50 White St, New York, NY.

Additional meetings of the club have been scheduled as follows:

October 9—"Modern Pigment Dispersion Methods", Hugh F. Purcell, Morehouse-Cowles Inc

November 13—"Practical Applications of Submicron Crystal Pigments", John H L Watson, M W Freeman Co.

December 11—"The Special Factors Influencing Carbon Black Dispersions", Louis J Venuto, Columbian Carbon Co.

January 8—"New Developments in Corrosion - Inhibitive Pigments", R L Whitney and R P Bates, National Lead Co.

Southern Latex to Represent Alco in South

Alco Oil & Chemical Corp, Philadelphia, Pa, and Southern Latex Corp, Madison, NJ, Austell, Ga, and Concord, NC, have announced that the latter will sell, distribute, and provide technical service on Alco's thickeners and chemicals in the Southeastern States.

Southern Latex will maintain adequate stocks of Alco's Alcogum thickeners, Vulcacure accelerators and antioxidants, and warp sizes at Concord, NC, and Austell and Dalton, Ga. In most instances, Southern Latex will make shipment in trucks operated by it.

• NAMES IN THE NEWS •

Estes

Billy G Estes has joined the technical sales staff of Crown Chemical Corp. He will cover portions of South Carolina, Georgia, Alabama and Tennessee.

Mr Estes, prior to joining Crown Chemical, was associated with J P Stevens and Company, Inc for several years in South Carolina and most recently with United Merchants and Mfrs, Inc, Old Fort, NC.

He will make his headquarters in Greenville, SC.

Francis K Burr, senior associate of Fabric Research Laboratories, Inc, Dedham, Mass, has been named General Chairman of the Wesleyan University Alumni Fund for 1958-59.

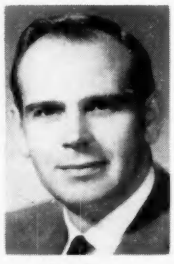
C Burke Miles has been named assistant research director of Food Machinery and Chemical Corp's Inorganic Chemicals Research and Development Department. Other appointments in the current move to enlarge and strengthen FMC's research set-up include the naming of **Robert L McEwen** as manager of development and **Fred R Sheldon** as general manager of the Applications Section.

Dr Miles for the past four years has directed the research activities of the Westvaco Mineral Products Division.

Before his present appointment, Mr McEwen was manager of the Central Development Department of FMC's Chemical Divsions.

Mr Sheldon's previous experience was with Becco Chemical Div, where he has held various research and sales service positions.

FMC's Inorganic Chemicals Department includes the Westvaco Chlor-Alkali Division, the Westvaco Mineral Products Division, and the Becco Chemical Division.



Isenburg

O E Isenburg, general manager of the Harmon Colors Division of B F Goodrich Chemical Company, has been elected president of the Dry Colors Manufacturers' Association. He previously served two years as vice president of the organization.

Mr Isenberg has been general manager of the Harmon operation since May, 1954, directing all operations of the company's color pigment business.

Bruce O Dickison has joined Carbic Color & Chemical Co Inc in the New England area. He will work out of the Providence office.

Prior to joining Carbic, he was with Arnold, Hoffman & Co in the Midwest.